TEXT FLY WITHIN THE BOOK ONLY

UNIVERSAL
LIBRARY

OU_158432

UNIVERSAL
LIBRARY

OSMANIA UNIVERSITY LIBRARY

Call No. 805.4/A615A Accession No. 96374

Author Abraham H.

Title Asphalts.

This book should be returned on or before the date last marked below.



Copyrighted by The American Museum of Natural History, New York, N. Y.

FIG. 1.—"The Death Trap of Rancho-la-Brea, California."

Painted by Charles R. Knight.

ASPHALTS
AND
ALLIED SUBSTANCES

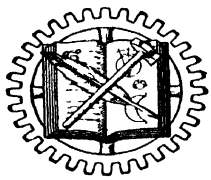
Their Occurrence, Modes of Production, Uses in the Arts and Methods of Testing

BY

HERBERT ABRAHAM

President, The Ruberoid Co.; President, Asphalt Shingle and Roofing Industry

FOURTH EDITION



NEW YORK

D. VAN NOSTRAND COMPANY, INC.

250 FOURTH AVENUE

Copyright, 1918 and 1920,
BY
D. VAN NOSTRAND COMPANY

Copyright, 1929, 1938 by
D. VAN NOSTRAND COMPANY, INC.

All rights reserved. No part of this book may be reproduced in any form without permission in writing from the publisher, except by a reviewer who may quote brief passages in a review to be printed in a magazine or newspaper.

First Published, August 1918

Second Edition, August 1920

Third Edition, November 1929
Reprinted, April 1932

Fourth Edition, January 1938

PRINTED IN U. S. A.

PRESS OF
BRAUNWORTH & CO., INC.
BUILDERS OF BOOKS
BRIDGEPORT, CONN.

PREFACE TO FOURTH EDITION

The present edition of "Asphalts and Allied Substances" has been revised in several material respects. Although in the main, the textual sequence of the earlier editions has been followed, the subject matter has been amplified so as to cover the ground more intensively from the earliest dawn of civilization, down to the very last word in this rapidly growing branch of technology.

Several new chapters have been added. The section relating to "Methods of Testing" has been completely rewritten to conform with the most recent practices. References to patents and to the general literature have been brought up to date, and more than 12,000 such citations have been segregated in an appendix at the end of the volume, where they would not interfere with the continuity of the text and yet be readily accessible to the interested reader. The "Bibliography" has likewise been expanded to embrace more than 900 treatises.

No effort has been spared to make this new edition as comprehensive as the limitations of space and the author's available time have permitted.

HERBERT ABRAHAM.

NEW YORK CITY,
September 15, 1937.

PREFACE TO THIRD EDITION

During the decade which has elapsed since the publication of the Second Edition of "Asphalts and Allied Substances," the technology of bituminous substances has made great strides under the pressure of rapidly increasing production. The output of crude petroleum has not only increased by leaps and bounds, but has gravitated towards oil-fields yielding a greater percentage output of asphalt. Similarly, coal tar and coal-tar pitch have been produced in larger amounts, because of the widespread introduction of equipment for recovering these valuable by-products.

Accordingly, the supply of petroleum asphalt and coal-tar residuals has more than kept pace with their utilization in the arts, and this has been reflected by a substantial reduction in their market price. Not only has this condition rendered these substances available to a larger consuming field, but it has also served to stimulate inventors to adapt them to new uses.

The increasing commercial importance of bituminous substances has been evidenced by greater efforts to perfect and standardize their methods of test, through such agencies as The Federal Specification Board (organized by the United States Government), and many scientific societies, including especially the American Society for Testing Materials, through whose courtesy the author has been given permission to reprint their specifications and tests in this book.

To keep pace with this rapidly developing art, it has accordingly been necessary to rewrite the book completely, and thereby bring it up-to-date.

HERBERT ABRAHAM.

NEW YORK CITY,
November 1, 1929.

PREFACE TO FIRST EDITION

This treatise has been written for those interested in the fabrication, merchandising and application of bituminous products. It embraces: (1) methods serving as a guide for the works chemist engaged in testing and analyzing raw and manufactured products; (2) data for assisting the refinery or factory superintendent in blending and compounding mixtures; (3) information enabling the ambitious salesman to enlarge his knowledge concerning the scope and limitations of the articles he vends; and (4) the principles underlying the practical application of bituminous products for structural purposes, of interest to the engineer, contractor and architect. Subject-matter of sole value to the technical man has been segregated in Part V, "Methods of Testing," excepting the outline of the "Chemistry of Bituminous Substances" appearing in Chapter III. These sections, however, may be passed over by the non-technical man, without interfering materially with the continuity of the work.

In view of the vast amount of ground covered in this volume, and fully realizing the limitations of his proficiency in some of its branches and ramifications, the author has taken it upon himself to draw freely from contemporary text-books and journal articles. In such instances, his endeavor has been to place due credit where it belongs, by referring to the source of such extraneous information. Nevertheless, there has been included a substantial amount of original data accumulated by the author during the past nineteen years, most of which appears in print herein for the first time.

Topics which have been ably presented in other reference books, as for example the technology of pavements, etc., have purposely been subordinated to those concerning which little data have hitherto been available. To the latter belong such subjects as petroleum asphalts; fatty-acid pitches; bituminized roofings, floorings and other fabrics; bituminous paints, cements, varnishes and japans.

Certain branches of the industry have developed along different lines in Europe than has been the case in this country, especially

the treatment of peat and lignite (Chapter XV), also pyrobituminous shales (Chapter XVI). In such instances, the methods in vogue abroad before the great war are described with more or less detail. It must be borne in mind in this connection, that the war has materially interfered with the prosecution of these industries abroad, and the data presented should be so construed, even though not specifically stated in the text.

Whereas the greatest pains have been taken to establish the accuracy of every assertion, as well as the authenticity of every alleged fact, the author does not flatter himself that he has escaped the pitfalls which must perforce beset the path of a writer who undertakes to delve into a subject as complicated as the one under consideration, concerning which there are so many divergent views.

Appreciation is expressed for the valuable suggestions and assistance rendered by W. A. Hamor, D. R. Steuart, Prévost Hubbard, S. R. Church, E. B. Cobb, David Wesson, Clifford Richardson, S. C. Ells, and the author's immediate associates.

HERBERT ABRAHAM.

NEW YORK CITY,
July 1, 1918.

TABLE OF CONTENTS

PART I—GENERAL CONSIDERATIONS

CHAPTER I

PAGE

HISTORICAL REVIEW 1

Origin of the Words "Asphalt," "Bitumen" and "Pitch"—Fossils Preserved by Means of Asphalt—Use of Asphalt by the Sumerians (about 3800 to 2500 B.C.)—*King Sargon of Accad* (*about* 3800 B.C.)—*Manishtusu King of Kish* (*about* 3600 B.C.)—*Ornaments* (*about* 3500 B.C.)—*At Tell-Asmar in Eshunna* (3200 *to* 2900 B.C.)—*Lugal-daudu King of Adab* (*about* 3000 B.C.)—*Entemena of Shirpula* (*about* 2850 B.C.)—*Ur-Ninâ King of Lagash* (*about* 2800 B.C.)—*Ornaments and Sculptured Objects* (2800 *to* 2500 B.C.)—*Gudea of Lagash* (*about* 2700 B.C.)—*Tablets of Gilgamish* (*about* 2500 B.C.)—*Bur-Sin King of Ur* (*about* 2500 B.C.)—Use of Asphalt by Pre-historic Races in India (about 3000 B.C.)—Use of Asphalt by the Early Egyptians (2500 to 1500 B.C.)—Use of Asphalt in Biblical Times (2500 to 1500 B.C.)—Use of Asphalt by the Babylonians (2500 to 538 B.C.)—*King Khammurabi* (*about* 2200 B.C.)—*Queen Semiramis* (*about* 700 B.C.)—*King Nabopolassar* (625 *to* 604 B.C.)—*King Nebuchadnezzar* (604 *to* 561 B.C.)—Use of Asphalt by the Assyrians (1400 to 607 B.C.)—*King Adad-Nirari I* (*about* 1300 B.C.)—*King Tukulti-Ninurta II* (890 *to* 884 B.C.)—*King Sargon* (722 *to* 705 B.C.)—*King Sennacherib* (704 *to* 682 B.C.)—Use of Asphalt in Constructing Lake-Dwellings (about 1000 B.C.)—References to Bituminous Substances by Greek and Roman Writers (500 B.C. to 817 A.D.)—*Herodotus* (484-425 B.C.)—*Thucydides* (471-401 B.C.)—*Hippocrates* (460-377 B.C.)—*Xenophon* (430-357 B.C.)—*Aristotle* (384-322 B.C.)—*Theophrastus* (372-288 B.C.)—*Antigonus* (*about* 311 B.C.)—*Hannibal* (247-183 B.C.)—*Vergil* (70-19 B.C.)—*Strabo* 63 B.C.—24 A.D.)—*Diodorus Siculus* (*about* 50 A.D.)—*Virtuvius* (*about* 50 A.D.)—*Pliny the Elder* (23-79 A.D.)—*Josephus Flavius* (37-95 A.D.)—*Plutarch* (*about* 46 A.D.)—*Tacitus* (55-117 A.D.)—*Aelian* (*Aelianus Claudius*) (*about* 100 A.D.)—*Dioscorides* (*about* 150 A.D.)—*Dion Cassius* (155-230 A.D.)—*Philostratus* (*The Elder*) (*about* 200 A.D.)—*Geoponica* (200-300 A.D.)—*Africanus* (*about* 300 A.D.)—*Ammianus Marcellinus* (330-395 A.D.)—*Theophanes* (758-817 A.D.)—About 950 A.D., Abu-L-Hasan Masudi—About 985 A.D., Abd Al Mukaddasi—About 1248 A.D., Pierre de Joinville—About 1300 A.D. Marco Polo—About 1350 A.D., Sir John Mandeville—1494-1555, Georg Agricola—1498, Christopher Columbus—About 1500, Use of Asphalt in Peru—1535, Discovery of Asphalt in Cuba—1563, Cesar Fredericke—1595, Sir Walter Raleigh—1599, First Classification of Bituminous Substances—1608, William Shakespeare—1656, Early Dictionary Definition of "Bitumen"—1660, John Milton—1661, Commercial Production of Wood Tar—1672, First Accurate Description of Persian Asphalt Deposits—1673, Discovery of Elaterite—1681,

PAGE

Discovery of Coal Tar and Coal-Tar Pitch—1691, Discovery of Illuminating Gas from Coal—1694, Discovery of Shale Tar and Shale-tar Pitch—1712-1730, Discovery of Val de Travers, Limmer and Seyssel Asphalt Deposits—1722, First Use of Tar for Flat Roofs—1746, Invention of the Process of Refining Coal Tar—1752, Samuel Foote—1777, First Exposition of Modern Theory of the Origin of Asphalt—1788, Discovery of Lignite Tar—1780-1790, Discovery of "Composition" or "Prepared" Roofing—1792-1802, Manufacture of Coal Gas and Coal Tar on a Large Scale—1797-1802, Exploitation of Seyssel Asphalt in France—1815, Commercial Exploitation of Coal-tar Solvents—1820, Manufacture of Asphalt-saturated Packing Papers in Switzerland—1822, Discovery of Scheereite and Hatchettite—1830, Discovery of Paraffin Wax—1832, Coal-tar First Used for Paving—1833, Discovery of Ozokerite—1835, First Asphalt Mastic Foot Pavements Laid in Paris—1836, Asphalt First Used in London for Foot Pavements—1837, Publication of First Exhaustive Treatise on the Chemistry of Asphalt—1837, Discovery of Bituminous Matter in the United States—1838, Discovery of Process for Preserving Wood with Coal-tar Creosote—1838, Asphalt First Used in the United States for Foot Pavements—1841, First Use of Wood Block Pavement—1843, Bituminous Matters Discovered in New York State—1844-1847, First Composition Roofing in the United States—1850, Discovery of "Asphaltic Coal" in New Brunswick, Nova Scotia—1852, First Modern Asphaltic Road—1854, First Compressed Asphalt Roadway Laid in Paris—1858, First Modern Asphalt Pavement Laid in Paris—1863, Discovery of Grahamite in West Virginia—1869, The First Compressed Asphalt Pavement in London—1870-1873, First Asphalt Roadways in the United States—1876, First Trinidad Asphalt Pavement Laid in the United States—1880, Use of Asphalt "Chewing-gum" in Mexico—1881, Use of Chemicals for Oxidizing Coal Tars and Petroleum Asphalts—1885, Discovery of Uintaite (Gilsonite) in Utah—1889, Discovery of Wurtzilite in Utah—1891, Exploitation of the Bermudez Asphalt Deposit, Venezuela—1892, Use of Bermudez Asphalt on a Large Scale—1894, Use of Air for Oxidizing Petroleum Asphalt.

CHAPTER II

TERMINOLOGY AND CLASSIFICATION OF BITUMINOUS SUBSTANCES 51

Bituminous Substances—Bitumen—Pyrobitumen—Petroleum—Mineral Wax—Asphalt—Asphaltite—Asphaltic Pyrobitumen—Non-asphaltic Pyrobitumen—Tar—Pitch—Classification of Bituminous Substances.

CHAPTER III

CHEMISTRY OF BITUMINOUS SUBSTANCES..... 65

Composition of Non-mineral Matrix—Composition of Associated Mineral Constituents—Composition of Associated Non-mineral Constituents—Behavior with Solvents—Behavior on Subjecting to Heat—Reactions with Gases — *Oxygenation* — *Hydrogenation* — Reactions with Acids—*Liquid Sulfur Dioxide—Sulfuric Acid and Sulfur Trioxide—Nitric Acid—Sulfuric Acid and Formaldehyde*—Reactions with Alkalies—Reactions with Metalloids—*Sulfur and Sulfur Dichloride—Selenium—Phosphorus—Halogens*—Reactions with Metallic Salts.

CHAPTER IV

PAGE
GEOLOGY AND ORIGIN OF BITUMENS AND PYROBITUMENS 82

Geology—*Age of the Geological Formations—Character of the Associated Minerals—Modes of Occurrence*—Springs—Lakes—Seepages—Subterranean Pools or Reservoirs—Impregnated Rock in Strata—Filling Veins—**Movement of Bitumen in the Earth's Strata**—*Hydrostatic Pressure—Gas Pressure—Capillarity—Gravitation—Effect of Heat*—**Origin and Metamorphosis of Bitumens and Asphaltic Pyrobitumens**—*Probable Origin of Bitumens and Asphaltic Pyrobitumens*—Inorganic Theories—Vegetable Theories—Animal Theories—*Metamorphosis of Mineral Waxes, Asphalts, Asphaltites and Asphaltic-Pyrobitumens from Petroleum*—**Origin and Metamorphosis of Non-Asphaltic Pyrobitumens.**

CHAPTER V

ANNUAL PRODUCTION OF BITUMINOUS SUBSTANCES AND THEIR MANUFACTURED PRODUCTS 98

Production of Asphalts, Asphaltites and Asphaltic Pyrobitumens—World Production—Production in the United States—**Production of Tars and Pitches**—Tars Derived from Coal—Tars and Pitches Derived from Wood—Other Tars and Pitches—**Manufactured Products**—Bituminous Paving Materials—Bituminous Roofing Products—Asphalted Felt-base Floor Coverings.

PART II—SEMI-SOLID AND SOLID BITUMENS AND PYROBITUMENS

CHAPTER VI

METHODS OF MINING, TRANSPORTING AND REFINING..... 112

Mining Methods—Open-cut Quarrying—Tunnelling—Special Methods—**Methods of Shipment and Transportation—Methods of Refining**—Dehydration — Distillation — Comminution — Sedimentation — Extraction —*Extraction by Means of Water—Extraction or Precipitation with Solvents*—**Methods of Storage.**

CHAPTER VII

MINERAL WAXES 129

Ozokerite—Europe—Galicia—Rumania—Russia—*Terek Province—Kuban Province—Kutais Province—Tiflis Province—Baku Province—Kars Province* —**Asia**—*State of Turkestan—Siberia—Philippine Islands*—**North America** —United States—*Utah—Texas*—**Hatchettite or Hatchettine—Scheereite Kabaite—Montan Wax.**

CHAPTER VIII

PAGE

NATIVE ASPHALTS OCCURRING IN A FAIRLY PURE STATE..... 139

North America—*United States*—Kentucky—Oklahoma—Utah—California—Oregon—*Mexico*—State of Tamaulipas—State of Vera Cruz—*Cuba*—Province of Matanzas—Province of Santa Clara—Province of Camaguey—Province of Santiago de Cuba—**South America**—*Venezuela*—State of Bermudez—State of Zulia—**Europe**—*France*—Department of Puy de Dôme—*Albania*—Sélénitza — *Greece* — Zante — *Russia*—Kutais Province—Tiflis Province—Uralsk Province—**Asia**—*Syria*—Villayet of Beirut—*Mesopotamia* (*Iraq*)—Hit—Ain el Maraj—Ain Ma' Moura—Quijarah, Ramadi and Abu Gir—*Asiatic Russia*—Sakhalin—*Philippine Islands*—Island of Leyte.

CHAPTER IX

NATIVE ASPHALTS ASSOCIATED WITH MINERAL MATTER..... 155

North America—*United States*—Kentucky—Missouri—Indiana—Oklahoma—Arkansas — Alabama — Louisiana — Texas — Utah — Wyoming — California—*Canada*—Alberta Province—Manitoba Province—*Mexico*—States of Vera Cruz and Tamaulipas—*Cuba*—Province of Matanzas—Province of Pinar del Rio—Province of Havana—Province of Camaguey—Province of Santiago de Cuba—**South America**—*Trinidad*—*Brazil*—State of Paraná—State of São Paulo—*Argentina*—Province of Jujuy—Province of Chubut—Province of Mendoza—*Colombia*—Department of Bolivar—Department of Antioquia—Department of Santander—Department of Boyacá—*Equador*—Province of Guayas—**Europe**—*France*—Department of Landes—Department of Gard—Department of Haute-Savoie—Department of Ain—Department of Basses-Alpes—Department of Puy-de-Dôme—Department of Haute Vienne—*Switzerland* — *Alsace-Lorraine* — *Spain* — Burgos Province — *Germany*—Province of Hanover—Province of Westphalia—Province of Hessen—Province of Baden—Province of Silesia—*Jugoslavia*—Province of Dalmatia—Province of Herzegovina—Province of Styria—*Austria*—Province of Tyrol—*Hungary*—Province of Bihar—*Czecho-Slovakia*—Province of Trencsen—Provinces of Moravia and Silesia—*Rumania*—*Albania*—Sélénitza—*Italy*—Compartment of Marches—Compartment of Abruzzi e Molise—Compartment of Calabria—Compartment of Campania—Compartment of Sicily—*Greece*—Department of Triphylia—Department of Achaia—Iolian Islands—Department of Phocis—Department of Phthiotis—Departments of Eurytania and Arta—Northern Departments—*Portugal*—Province of Estremadura—*Spain*—Province of Santander—Province of Alava—Province of Navarre—Province of Gerona—Province of Tarragona—Province of Soria—Province of Burgos—Province of Almería—Province of Valencia—*Russia* (*in Europe*)—Simbirsk Province—Kazan Province—Samara Province—Terek Province—Kutais Province—Tiflis Province—Baku Province—**Asia**—*Syria* (*Levant States*)—Vilayet of Aleppo—Vilayet of Beirut—Vilayet of Sham—Palestine—*Mesopotamia* (*Iraq*)—Valiyet of Bagdad—*Asiatic Russia*—Uralsk Province—State of Turkestan—Kamchatka Peninsula—Sakhalin Island—*Turkey-in-Asia* (*Asia Minor*)—Anatolia—*Arabia*—Vilayet of El Hasa—Sinai Peninsula—*Egypt*—*India*—Kashmir District—Hazara District—Beluchistan District—Bombay Island—*China*—Chinese Turkestan—*Japan*—Akita Prefecture—Prefectures of Yamagata, Aomori and District of Hokkaido—*Australia*—New South Wales—Western Australia—North-

PAGE

ern Territory—Tasmania—New Zealand—*Dutch East Indies*—Buton (Boeten) Island—**Africa**—*Algeria*—Province of Oran—*Nigeria*—*Rhodesia*—*Madagascar.*

CHAPTER X

ASPHALTITES 227

GILSONITE or UINTAITE—**North America**—*United States*—Utah—Oregon—**Asia**—*Russia*—Archangel Province—GLANCE PITCH—**North America**—*West Indies*—Barbados—Santo Domingo (Haiti)—*Cuba*—Province of Pinar del Rio—Province of Santa Clara—Province of Camaguey—*Mexico*—State of Vera Cruz—*United States*—Utah—**Central America**—*Nicaragua*—District of Chontales—*Salvador*—Department of San Miguel—**South America**—*Colombia*—Province of Tolima—Province of Bolivar—**Europe**—*Germany*—Bentheim—*Syria* (*Levant States*)—Vilayet of Sham—Palestine—*Mesopotamia* (*Iraq*)—GRAHAMITE—**North America**—*United States*—West Virginia—Texas—Oklahoma—Colorado—*Mexico*—State of Vera Cruz—State of San Luis Potosi—State of Tamaulipas—*Cuba*—Province of Pinar del Rio—Province of Havana—Province of Santa Clara—**South America**—Trinidad—*Argentina*—Province of Mendoza—Province of Neuquen—*Peru*—Province of Tarma.

CHAPTER XI

ASPHALTIC PYROBITUMENS 262

ELATERITE—*England*—Derbyshire County—*Australia*—State of South Australia—*Asiatic Russia*—State of Turkestan—WURTZILITE—*United States*—Utah—**Albertite**—*Canada*—Province of New Brunswick—Province of Nova Scotia—*United States*—Utah—*South America*—Falkland Islands—*Germany*—Province of Hanover—*Australia*—Tasmania—*Portuguese West Africa*—Province of Angola—IMPSONITE—**North America**—*United States*—Oklahoma—Arkansas—Nevada—Michigan—**South America**—*Peru*—Provinces of Canta and Yauli—Province of Huarochiri—*Brazil*—State of São Paulo—*Australia*—West Australia.

CHAPTER XII

PYROBITUMINOUS SHALES 275

PART III—TARS AND PITCHES

CHAPTER XIII

GENERAL METHODS OF PRODUCING TARS..... 278

Destructive Distillation—Composition of the Substance—The Temperature—The Time of Heating—The Pressure—The Efficiency of the Condensing System—**Partial Combustion with Air and Steam**—**Partial Combustion with a Limited Access of Air**—**Cracking of Oil Vapors.**

CHAPTER XIV

PAGE

WOOD TAR, WOOD-TAR PITCH AND ROSIN PITCH..... 288

Wood Tar and Wood-tar Pitch—Varieties of Wood Used—Yields of Distillation—*Hardwood Distillation*—Method of Distilling—Refining Processes—*Soft (Resinous) Wood Distillation*—Method of Distilling—Refining Processes—Hardwood Tar and Pine Tar—Hardwood-tar Pitch and Pine-tar Pitch—**Rosin Pitch**—Raw Materials Used—Methods of Distilling—Products Obtained—Properties of Rosin Pitch—"Burgundy Pitch."

CHAPTER XV

PEAT AND LIGNITE TARS AND PITCHES..... 305

Peat Tar and Peat-tar Pitch—Formation of Peat—Varieties of Peat—Methods of Collecting—Dehydrating Processes—Methods of Distilling—Refining Processes—Properties of Peat Tar—Properties of Peat-tar Pitch—**Lignite Tar and Lignite-tar Pitch**—Varieties of Lignite—Mining Methods—Methods of Distilling—Products Obtained—Treating Impure Lignite—Properties of Lignite Tar—Refining Processes—Products Obtained—Properties of Lignite-tar Pitch.

CHAPTER XVI

SHALE TAR AND SHALE-TAR PITCH..... 324

Shale Mining—Retorts Used for Distillation—*Pumpherston Retort—Henderson or Broxburn Retort*—Methods of Recovering Shale Tar—Products Obtained—Properties of Shale Tar—Refining of Shale Tar—Properties of Shale-tar Pitch.

CHAPTER XVII

COAL TAR AND COAL-TAR PITCH..... 336

Bituminous Coals Used—Temperature of Treatment—*Production of Gas-Works Coal Tar*—Retorts Used—Methods of Recovery—Products Obtained—*Production of Coke-oven Coal Tar*—Retorts Used—Methods of Recovery—Products Obtained—*Production of Blast-furnace Coal Tar*—Methods of Recovery—*Production of Producer-gas Coal Tar*—Products Obtained—*Production of Low-temperature Tars*—Products Obtained—*Properties of Coal Tars—Methods of Dehydrating Coal Tar*—Settling—Use of Centrifuges—Horizontal Stills—Tube Heaters—Cascade System—*Methods of Distilling Coal Tar*—Simple Batch Stills—Vacuum Distillation—Steam Distillation—Gas Recirculation—Continuous Stills—Tube Stills—Collector-Main Condensers—*Recovery and Treatment of Coal-tar Distillates—Recovery and Treatment of Coal-tar Residuals*—Commercial Varieties of Coal-tar Pitch—Pitch Coke—*Properties of Coal-tar Pitches*—Gas-works Coal-tar Pitches—Coke-oven Coal-tar Pitch—Blast-furnace Coal-tar Pitch—Gas-producer Coal-tar Pitch—Low-temperature Coal-tar Pitch—Anthracene Pitch—Naphthol Pitch—Cresol Pitch.

CHAPTER XVIII

PAGE

WATER-GAS AND OIL-GAS TARS AND PITCHES..... 378

Carburetted Water-gas Tar—Method of Production—Properties of Water-gas Tar—*Oil-gas Tars*—Methods of Production—Pintsch Gas—Oil-water Gas—Blau Gas—Properties of Oil-gas Tars—*Refining of Water-gas and Oil-gas Tars—Properties of Water-gas-tar Pitch and Oil-gas-tar Pitch.*

CHAPTER XIX

FATTY-ACID PITCH, BONE TAR AND BONE-TAR PITCH..... 386

Fatty-acid Pitch—Sources from which Obtained—*Production of Candle and Soap Stocks*—Hydrolysis by Means of Water—Hydrolysis by Means of Concentrated Sulfuric Acid—Hydrolysis by the "Mixed Process"—Hydrolysis by Means of the Sulfo-compounds—Hydrolysis by Means of Ferments—*Refining Vegetable Oils by Means of Alkali*—Refining of Cotton-seed Oil—Refining of Corn-Oil—*Refining Refuse Greases*—Refining Packing-house and Carcass-rendering Greases—Refining Bone Grease—Refining Garbage and Sewage Greases—Refining Woolen-mill Waste—*Treatment of Wool Grease—Physical and Chemical Properties of Fatty-acid Pitches*—**Bone Tar and Bone-tar Pitch**—Methods of Production—Physical and Chemical Characteristics—Glycerine Pitch.

PART IV—PYROGENOUS ASPHALTS AND WAXES

CHAPTER XX

PETROLEUM ASPHALTS 409

Varieties of Petroleum—Asphaltic Petroleums—Semi-asphaltic Petroleums—Non-asphaltic Petroleums—**Dehydration of Petroleum**—Settling—Centrifuging—Tube-stills—**Distillation of Petroleum**—Batch Stills—*Steam Distillation—Dry Distillation*—Continuous Stills—Tube or Pipe Stills—**Cracking Processes**—Liquid Phase Cracking—*Tube-and-tank Process—Cross Process—Holmes-Manly Process*—Mixed Phase Cracking—*Burton Process—Dubb's Process*—Liquid Products—Semi-solid to Solid Distillates—Semi-solid to Solid Residues—**Residual Oils**—Varieties Obtained—Physical and Chemical Characteristics—**Blown Petroleum Asphalts**—Processes Used—Advantages of "Blowing" Over the Steam-distillation Process—Physical and Chemical Characteristics—**Sulfurized Asphalts**—**Residual Asphalts**—Processes Used—Physical and Chemical Characteristics—Methods of Distinguishing between Petroleum Asphalts and Native Asphalts—**Sludge Asphalts**—Methods of Production—Physical and Chemical Characteristics.

CHAPTER XXI

PARAFFIN WAX, WAX TAILINGS AND RESINS..... 466

Paraffin Wax—Sources from which Obtained—Physical and Chemical Characteristics—**Wax Tailings**—Methods of Production—Physical and Chemical Characteristics—**Petroleum Resins—Asphaltic Resins.**

CHAPTER XXII

PAGE

WURTZILITE ASPHALT 472

Method of Production—Still Used—Depolymerization Process—Grades of Wurtzilite Asphalt Produced—Chemical and Physical Characteristics.

PART V—MANUFACTURED PRODUCTS AND THEIR USES

CHAPTER XXIII

COMPOUNDING OF BITUMINOUS SUBSTANCES..... 476

General Considerations—Hardness, Fusibility, Penetration, Approximate Comparative Volatility, Weatherproof Properties and Efficiency of Fluxing of Various Bituminous Substances—Principles Involved in Preparing Mixtures —*Binary Mixtures*—Softening of the Substance and Lowering its Fusing-point—Augmenting the Adhesive Properties of the Substance—Increasing the Fluidity of the Substance when Melted—Effecting a More Perfect Union or Blending of the Constituents—Hardening the Substance, Raising its Fusing-point and Increasing its Stability—Rendering the Mixture Less Susceptible to Temperature Changes—Increasing the Tensile Strength of the Mixture—Making the Mixture More Weatherproof—Rendering Wax-like, Unctuous to the Feel, or Lessening the Tendency Towards Stickiness—*Tertiary and Complex Mixtures—Classes of Bituminous Mixtures*—Soft (Liquid) Bituminous Products—Medium (Semi-liquid to Semi-solid) Bituminous Products—Hard (Solid) Bituminous Products—*Processes of Compounding Bituminous Substances.*

CHAPTER XXIV

BITUMINOUS SUBSTANCES ADMIXED WITH DISCRETE AGGREGATES 494

Methods of Incorporating Discrete Aggregates—**Colloidal Particles**—*Admixed Mechanically*—Inorganic—Organic—*Liberated "In Situ"*—Inorganic —Organic—**Fillers (Powders) Including Pigments**—*Inorganic*—Oxides—Silicates—Carbonates—Sulfates—Phosphates—Miscellaneous Rock Products —Pyrogenous Products—Black Pigments—Colored Pigments—*Organic*—Vegetable Products—Black Pigments—**Fibers**—Inorganic—Organic—**Granular Matter**—Inorganic—Organic—**Coarse Mineral Aggregates**—Crushed Rock, Stone, Gravel or Slag—Graded Aggregates—**Combinations of the Foregoing.**

CHAPTER XXV

BITUMINOUS SUBSTANCES DISPERSED IN WATER 503

Types of Bituminous Dispersions—Forms of Apparatus Used—Character of the Bituminous Substance—**Character of the Dispersing Agent**—*Inorganic Substances*—Hydroxides—Oxides—Silicates—Sulfates—Phosphates—Sulfides or Polysulfides—Alkalies—Miscellaneous—*Organic Substances*—Fats

PAGE

and Oils—Resins—Soaps—Sulfonated Vegetable Oils—Sulfite Liquor—Mineral-Oil Derivatives—Carbonaceous Matter—Alkaline Bases—Proteins or Proteids—Albumenoids—Pectins—Gums and Algae—Polysaccharides and Hemicelluloses—Tannins—Miscellaneous—*Various Combinations*—Clay in Combination—Sodium Silicate in Combination—Soaps in Combination—Sulfonated Vegetable Oils in Combination—Alkaline Caseinates in Combination—Glue or Gelatine in Combination—Tannic Acid in Combination—**Uses of Bituminous Dispersions.**

CHAPTER XXVI

BITUMINOUS SUBSTANCES DISSOLVED IN SOLVENTS..... 514

Classes of Solvents—Function of Solvents—Behavior of Solvents—Solubility of Bituminous Substances—Adsorption of Bituminous Substances.

CHAPTER XXVII

SOLID, SEMI-SOLID AND SEMI-LIQUID BITUMINOUS COMPOSITIONS. 526

Adhesive Compounds for Built-up Roofing and Waterproofing Work—Adhesive Compounds for Waterproofing—Below Ground—Above Ground—Adhesive Compounds for Built-up Roofing Work—*Plastic-slate Cement—Calking Compounds—Bituminous Grout—Bituminous Enamel for the Inside of Steel Ships*—Bituminous Enamel—Bituminous Primer—*Bituminous Enamel for Acid-proofing Concrete Surfaces—Pipe Dips and Pipe-sealing Compounds*—Pipe Dips—Pipe Wrappings—Pipe-sealing Compounds—*Electrical Insulating Compounds*—Vacuum Impregnating Compounds—Cable-splicing and Pothead Compounds—Battery-box Compounds—"Carbons" for Batteries, Electric Lights and Armature Brushes—*Bituminous Rubber Substitutes—Molded Compositions*—Mixtures for Small Molded Articles—Preformed Joints and Washers—Molded Brake-linings, Clutch-facings and Friction Elements—Bituminated Cork Mixtures—Battery Boxes—Fibrated Bituminous Compositions—Roof Tiles—Floor Tiles—Floor and Step Treads—Artificial Lumber and Railroad Ties—Pipes and Conduits—Burial Vaults—Phonograph Records—Bituminated Leather Mixtures—Briquette Binders—Core Compounds—*Miscellaneous Bituminous Products*—Bituminous Fuels—Tars and Oils for the Flotation of Ores—*Waterproofing Compounds for Portland-cement Mortar and Concrete*—Pure Bituminous Substances—Bituminous Substances in Emulsified Form—Methods of Use—*Sponge Asphalt—Asphalt Dust and Filaments—Asphalt Jelly.*

CHAPTER XXVIII

BITUMINOUS PAVING MATERIALS..... 567

Bituminous Binders—Liquid Asphaltic Binders—Semi-solid to Solid Asphaltic Binders—Coal-tar Binders—Cut-back Products—Bituminous Emulsions—**Mineral Aggregates—Bituminous Compositions for Dust-laying (Cold Application)**—Bituminous Emulsions—Non-emulsified Products—General Considerations—**Bituminous Surfacings**—*Surface Treatment*—Silt Roads—Clay Roads—Sandy Roads—*Seal-Coats*—To Loose Surfaces—To Bonded Surfaces—To Old Bituminous Surfaces—To Non-bituminous Surfaces—*Road-Mix Wearing Course*—Open Aggregate ("Macadam Type")—

PAGE

Dense Aggregate ("Graded Type")—*Plant-Mix Wearing Course*—Open Aggregate ("Macadam Type")—Dense Aggregate ("Graded Type")—**Bituminous Macadam Pavements**—Foundation—Base Course—Surface Course—*Bituminous Emulsions*—Penetration Method—Road-Mix Method—**Bituminous Concrete Pavements**—Foundation—Base Course ("Black-base")—Surface Course, Coarsely-graded Type—Surface Course, Finely-graded Type—Asphalt Emulsions—**Sheet-asphalt Pavements**—Foundation—Binder and Surface Courses—General Considerations—**Bituminous Joint-Fillers**—Filling the Joints—**Asphalt-block Pavements**—Foundation—Laying the Blocks—**Bituminized Wood-block Pavements**—Methods of Impregnation—Creosote Preservatives—Foundation—Cushion Layer—Filling the Joints—General Considerations—**Asphalt Mastic Foot-Pavements and Floors**—Asphalts Used—Methods of Preparation—Methods of Application—**Asphalt Mastic Roofs**—**Bituminous Expansion Joints**—Promolded Strips—Bituminized Fabric Strips—Laminated Strips—Armored Strips—**Bituminous Rail-fillers**—**Asphalt Bridge-planking**—**Bituminous Revetment.**

CHAPTER XXIX

BITUMINIZED FABRICS, FELTS AND PAPERS FOR ROOFING, FLOORING, WATERPROOFING, BUILDING AND INSULATING PURPOSES 617

Prepared Sheet Roofings—*Felted Fabrics*—Rag-felt—Methods of Fireproofing Rag-felt—String-felt or Threaded-felt—Asbestos Felt—*Woven Fabrics*—*Bituminous Saturating Compositions*—Asphaltic Products—Tar Products—*Bituminous Coating and Adhesive Compositions*—*Fillers*—Black Pigments—Colored Pigments—*Inorganic Surfacings*—Fine Mineral Particles—Moderately Coarse Mineral Granules—Coarse Mineral Granules—*Organic Surfacings*—**Saturated Felt**—Saturating the Fabric—Tarpaulins—Brattice Cloth—Tents and Awnings—Waterproof Membranes—Wrapping Cloth—Teredo-proof Coverings—Roof Coverings for Railway Cars—**Prepared Roll-roofings**—*Single-layered Prepared Roofings*—*Laminated Roofings*—*Decorative Roll-roofings*—*Scalloped and Serrated Roll-roofings*—**Asphalt Shingles**—*General Features*—Features Pertaining to Thickness—Shingles Surfaced with Mineral Granules—Rear Surface Coating—Shingles Surfaced with Mineral Granules and Coated with Hydraulic Cement—Shingles Surfaced with Slate-veneer or Tile or Asbestos-cement—Shingles Surfaced with Metals—Shingles Reinforced with a Core of Sheet-metal or Wire—Laminated Shingles—Combinations with Asbestos Felt—Combinations Involving the Use of Wooden Shingles—Miscellaneous Forms—*Individual Shingles*—Forming Rectangular Patterns when Laid—Forming Diamond-shaped Patterns when Laid—Forming Hexagonal Patterns when Laid—Forming Octagonal Patterns when Laid—Forming Circular Patterns when Laid—Forming Thatched Effects when Laid—Reversible Forms—*Strip Shingles*—Forming Rectangular Patterns when Laid—Forming Diamond-shaped Patterns when Laid—Forming Hexagonal Patterns when Laid—Forming Octagonal Patterns when Laid—Forming Circular or Curved Effects when Laid—Forming Thatched Effects when Laid—Forming Miscellaneous Patterns when Laid—Reversible Forms—*Asphalt Sidings*—*Fastening Devices*—Lap-cement—Nails—Metal Cleats for Roll-roofings—Concealed Nailing for Roll-roofings—Forming Standing Seams—*Methods of Packaging*—*Methods of Laying Roofings and Shingles*—Laying the Fabric in a Single Course—Built-up Roofs—Laying Asphalt-shingle Roofs—*Fire-resisting Properties of Prepared Roofings and Shingles*—Bi-

PAGE

tuminized Floor Coverings—Saturated Felt Base—Method of Printing—Waterproofing Membranes—Materials Used—Preparing the Underlying Surface—Selecting and Installing the Waterproofing Membrane—Protecting the Waterproofing Membrane—Insulating and Building Papers—Raw Paper Stock—Bituminous Saturation—Bituminous Coating Compositions—Method of Manufacture—Bituminized Papers for Electrical Insulation—Bituminized Papers for Wrapping and Packing Purposes—Bituminized Papers for Mulching Plants and Crops—Blasting Fuses—Bituminized Cords and Ropes—Insulation for Electrical Transmission Wires—Bituminized Fiber Conduits—Asphalt Pipes—Brake Linings and Clutch-facings for Automobiles—Insulating and Acoustical Felts for Automobiles—Electrical Insulating Tape—Bituminized Wall-Board—Bituminous Insulating Board—Acoustical Blocks—Bituminous Stucco-base and Plaster-board.

CHAPTER XXX

BITUMINOUS LACQUERS, CEMENTS, VARNISHES, ENAMELS AND JAPANS 720

Bituminous Lacquers and Paints—Nature of the Base—Nature of the Fillers and Pigments—Nature of the Solvents—Methods of Manufacture—*Types of Bituminous Paints*—Masonry Coatings—Clear Damp-proofing Paints—Black Damp-proofing Paints—Stone Backing—Paints for Resurfacing Prepared Roofings—Asphalt Fibrous-Roof-Coating—Acid-resisting Coatings—Bituminous Coatings for Metal or Wood—Anti-fouling Paints—Bituminous Emulsion Paints—Flooring Compositions for Cold Application—Bituminous Cements—Method of Manufacture—Method of Use—Bituminous Varnishes—Method of Manufacture—Bituminous Enamels—Method of Manufacture—Cellulose-Ester Lacquers—Bituminous Japans—Method of Manufacture—Uses.

PART VI—METHODS OF TESTING

CHAPTER XXXI

SAMPLING 757

General Methods—Definitions—Sampling Crude, Refined and Blended Bituminous Substances—*Sampling at Place of Manufacture*—When Material is Pumped under Pressure—When Materials Flow by Gravity—*Sampling at Point of Delivery*—Semi-solid or Solid Materials—Liquid Materials—Solid Bituminous Materials in Crushed Fragments or Powder—Sampling Bituminous Paving Materials—Sampling Bituminized Fabrics—Sampling Bituminous Lacquers, Cements, Varnishes and Japans, also Bituminous Emulsions.

CHAPTER XXXII

EXAMINATION OF CRUDE, REFINED AND BLENDED BITUMINOUS SUBSTANCES 776

Synoptical Table of Bituminous Substances—Physical Characteristics—*Color*—Color in Mass—Color in Solution—*Homogeneity*—To the Eye—Under Microscope—When Melted—In Solution—*Appearance Surface when Aged*—

PAGE

Fracture—Lustre—Streak—Water Absorption—Diffusibility—Specific Gravity —Hydrometer Method—Westphal Balance Method—Bottle Method—Pyknometer Method—Analytical Balance Method—*Voids (Entrapped Air)—Colloidal Capacity*—Clay Dispersions—Ultramicroscopic Count of Colloidal Particles—**Mechanical Tests**—Viscosity—Engler Method—Saybolt (Furol) Method — Absolute Viscosity — Hutchinson's Method — Float Test — Schutte Method—Falling Ball Method—Alternating Stress Method—Falling Coaxial Cylinder Method—*Hardness or Plasticity*—Moh's Scale—Penetrometer—Consistometer—Susceptibility Index—*Ductility*—Dow's Method—Author's Method —*Tensile Strength (Cohesiveness)*—Author's Method—*Adhesiveness*—Riehm's Method—Wedmore's Method—Brown's Method—*Surface Tension*—Nellensteyn's Method—Interfacial Tension—**Thermal Tests**—*Thermal Conductivity*—A.S.T.M. Method—*Specific Heat—Heat Content—Thermal Expansion*—A.S.T.M. Method—*Breaking Point*—Knife Test—Reeve and Yeager's Method—Fraas Method—*Solidifying-Point*—Metzger's Method—*Softening-Point or Fusing-Point*—Krämer-Sarnow Method—Ring-and-Ball Method —Cube Method—Compression Method—A.S.T.M. Method for Petrolatum—A.S.T.M. Method for Paraffin Wax—*Flow-Point*—Richardson's Method—*Liquefying-Point*—Ubbelohde's Method—*Twisting-Point*—Taylor's Method—*Volatile Matter*—A.S.T.M. Method—*Evaporation Test*—A.S.T.M. Method—*Distillation Test*—A.S.T.M. Method—*Flash-Point*—Pensky-Martens Tester—Cleveland Tester—Tag Closed Tester—Tag Open Tester—*Burning-Point—Fixed Carbon*—**Solubility Tests**—*Solubility in Carbon Disulfide*—Where the Constituents are Not to be Examined Further—Where the Constituents are to be Examined Further—*Carbenes*—Richardson's Method—*Solubility in Petroleum Naphtha—Insoluble in Benzol ("Free Carbon")—Solubility in Other Solvents*—**Chemical Tests**—*Water*—Substances Distilling at Low Temperatures—Substances Distilling at High Temperatures—*Elemental Composition*—Carbon—Hydrogen—Sulfur—Nitrogen—Oxygen (in Non-mineral Matter)—*Molecular Weight*—Freezing-point Method—*Tar Acids*—Contraction Method—Liberation Method—*Naphthalene—Solid Paraffins*—Holde's Method —*Sulfonation Residue*—Residue Insoluble in Concentrated Sulfuric Acid—Residue Insoluble in Water—Dimethyl Sulfate Method—*Formolite Reaction*—Nastjukoff Method—*Degree of Mercuration—Saponifiable Constituents*—Free Acids (Acid Value)—Lactones and Anhydrides (Lactone Value)—Neutral Fats (Ester Value)—Saponification Value—Separation of Saponifiable Constituents—Examination of Unsaponifiable Constituents—Examination of Saponifiable Constituents—Glycerol—*Asphaltic Constituents*—Free Asphaltous Acids—Asphaltous Acid Anhydrides—Asphaltenes—Asphaltic Resins—Oily Constituents—*Diazo Reaction*—Graefe's Method—*Anthraquinone Reaction—Liebermann-Storch Reaction*—Colorimetric Method.

CHAPTER XXXIII

EXAMINATION OF BITUMINOUS SUBSTANCES COMBINED WITH DISCRETE AGGREGATES 1017

Physical Tests of Finished Product—*Paving Compositions, Asphalt Mastic, Bituminous Grouts, Pipe-sealing Compounds, etc.*—Specific Gravity—Voids—Resistance to Moisture—Effect of Water on Adhesion—Hardness—Resistance to Displacement—Extrusion of Binder Under Pressure—Resistance to Impact—Brittleness or Shatter Test—Coefficient of Wear—*Molded Materials*—Thickness—Resistance to Moisture—Tensile Strength—Compressive Strength—

PAGE

Flexural Strength—Distortion Under Heat—Softening-Point—Resistance to Impact—Electrical Tests—**Separation of Finished Product into Its Component Parts—Separation of the Bituminous Matter and Discrete Aggregate**—*Methods Suitable for Aggregates Associated with an Asphaltic Binder*—Hot Extraction Method—Cold Extraction Method—Centrifugal Extraction Method—*Method Suitable for Aggregates Associated with Coal-tar Pitch Binder*—**Recovery and Examination of Extracted Bituminous Matter**—*Separation of Bituminous Constituents*—**Examination of the Separated Aggregate**—*Inorganic Aggregates*—Granularmetric Analysis—Elutriation Test—Air-separation Test—Adsorptive Capacity of Fine Fillers—Specific Gravity—*Organic Particles, Fibers, Fillers, etc.*

CHAPTER XXXIV

EXAMINATION OF BITUMINIZED FABRICS 1072

Physical Tests of the Finished Product—*Weight per Unit Area*—For Saturated Felted and Woven Fabrics—For Smooth-roll and Mineral-surfaced Roll-roofing—For Mineral-surfaced Shingles—*Thickness—Strength*—Tensile Strength—Bursting Strength—Tearing Strength—*Pliability*—Mandrel Test—Reeve and Yeager Test—*Resistance to Dampness—Water Absorption—Resistance to Heat*—For Asphalt-saturated Fabrics—For Coal-tar Saturated Fabrics Only—For Asphalt Roll-roofings and Shingles—*Electrical Tests—Special Tests Applicable to Insulating Tape*—Tensile Strength—Adhesion—Oven Test—Tackiness—**Separation of Finished Product into Its Component Parts—Separation of Bituminous Matter, Mineral Matter and Fibrous Constituents**—*Moisture—Analysis of Saturated Fabrics (Single-layered)—Analysis of Saturated and Coated Fabrics (Single-layered)*—**Recovery and Examination of Extracted Coatings and Saturation—Examination of the Separated Mineral Surfacing and Admixed Mineral Constituents—Examination of the Separated Fabric**—*Weight per Unit Area ("Number")*—Uncorrected Number—Moisture—Corrected Number—*Thickness—Tensile Strength—Porosity—Speed with which the Felt Will Saturate*—Vertical Method—Horizontal Method—*Saturating Capacity—Fiber Composition.*

CHAPTER XXXV

EXAMINATION OF BITUMINOUS SOLVENT COMPOSITIONS 1117

Physical Tests of the Finished Product—*Résumé of Physical Tests*—Specific Gravity—Viscosity—Plasticity and Mobility—Flash-point—Spreading Capacity and Workability—Draining Test—Time of Drying—Hiding Power—Color—Gloss—Hardness, Abrasion and Adhesion—Water Absorption—Resistance to Heat—Resistance to Oil—Resistance to Acids and Alkalies—Dielectric Strength—**Estimation, Recovery and Examination of the Solvent**—*Estimation and Recovery of Solvent*—Evaporation Method—Steam Distillation Method—*Examination of the Solvent*—**Estimation, Recovery and Examination of Pigment and Filler**—*Estimation and Recovery of Pigment and Filler—Examination of the Pigment or Filler*—**Estimation, Recovery and Examination of the Base**—*Estimation and Recovery of the Base—Examination of the Base.*

CHAPTER XXXVI

PAGE

EXAMINATION OF BITUMINOUS DISPERSIONS..... 1132

Physical Tests of the Finished Product—*Method of Identification*—*Homogeneity*—Appearance Under Microscope—Sieve Test—Settlement Test—Stability on Aging—*Viscosity*—*Demulsibility*—Calcium-chloride Test—Ferrous-sulfate Test—Behavior with Aggregate ("Coating Test")—*Miscibility with Water*—*Effects on Freezing*—*Resistance to Water After Setting*—Without Aggregate—When Mixed with Aggregate—**Separation of the Dispersion into its Component Parts**—Distillation Residue—Water and Volatile Oils—Dispersing Agents.

CHAPTER XXXVII

WEATHERING TESTS 1147

Effects of Weathering—Evaporation—Oxidation—Carbonization—Polymerization—Effects of Moisture—**Actual Weathering Test**—Testing Bituminized Fabrics—Testing Bituminous-solvent Compositions—Testing Crude, Refined or Blended Bituminous Substances—**Accelerated Weathering Test**—Testing Bituminized Fabrics—Testing Bituminous-solvent Compositions—Testing Bituminous Compositions—**Modified Accelerated Weathering Test.**

TEMPERATURE CONVERSION TABLE 1182

BIBLIOGRAPHY 1183

REFERENCES 1231

INDEX 1435

ASPHALTS AND ALLIED SUBSTANCES

PART I

GENERAL CONSIDERATIONS

CHAPTER I

HISTORICAL REVIEW

Origin of the Words "Asphalt," "Bitumen" and "Pitch." The word "ăsphalt" is claimed to have been derived from the Accadian term "asphaltu" or "sphallo," meaning "to split." It was later adopted by the Homeric Greeks in the form of the adjective ἀσφαλής, ές, signifying "firm," "stable," "secure," and the corresponding verb ἀσφαλιζω, ίσω, meaning "to make firm or stable," "to secure." It is a significant fact that the first use of asphalt by the ancients was in the nature of a cement for securing or joining together various objects, and it thus seems likely that the name itself was expressive of this application. From the Greek, the word passed into late Latin, and thence into French ("asphalte") and English ("asphalt").

The expression "bitumen" originated in the Sanskrit, where we find the words "jatu," meaning "pitch," and "jatu-krit," meaning "pitch creating," "pitch producing" (referring to coniferous or resinous trees). The Latin equivalent is claimed by some to be originally "gwitû-men" (pertaining to pitch), and by others, "pixtumens" (exuding or bubbling pitch), which was subsequently shortened to "bitûmen," thence passing via French into English. From the same root is derived the Anglo Saxon word "cwidu" (Mastix), the German word "Kitt" (cement or mastic) and the old Norse word "kvada."

The following terms have been traced throughout the various ancient languages:

Sumerian:

Esir (petroleum and native asphalt); esir-lah (hard, glossy asphalt); esir-harsag (rock asphalt); esir-é-a (mastic asphalt); še-li-ud (pine tar); kunin or esir-ud-du-a (pitch).

Sanskrit:

Jatu (native asphalt also pitch); śilā-jatu or aśmajātam-jatu (rock asphalt).

Assyrian and Accadian:

Iddū, ittû, it-tû-u, or amaru (native asphalt); kukru or kir-kir-anu (pine tar); sippatu or kupru or ku-pur (pitch). In Babylonia, pitch is still termed "kupru."

Hebrew:

Zephet or hêmâr (native asphalt); zephet or kopher or kofer (pitch). In Exodus (II, 3) we find the words "hêmâr" and "zephet" denoting pitch.

Arabic and Turkish:

Seyali or zift or zipht (native asphalt); chemal or humar (houmar) or gasat (qasat) (rock asphalt); ghir or gir or kir or kafr (mastic asphalt); zipht or kir or kafr (pitch); neftgil (mineral wax).

Greek:

Maltha (soft asphalt); asphaltos (native asphalt); pissasphaltos or pittasphaltos or pittolium (rock asphalt); pissa-hygra or pisselaion (pine tar); pissa or pitta (pitch); ampelitis (mineral wax and asphaltites); spinos (bituminous coal); anthrax (anthracite coal).

Latin:

Maltha or bitumen liquidum (soft asphalt); bitumen (native asphalt); pix liquida or serum picis (pine tar); pix (pitch); gagates (asphaltite and jet and lignite); lapis thracius (bituminous coal); carbo (anthracite coal).

Fossils Preserved by Means of Asphalt. One of the most interesting cases on record is in connection with the fossilized remains of plants and animals recovered from the Rancho-la-Brea asphalt pits in Los Angeles County, California, about eight miles from the City of Los Angeles.[1] These consist of a series of crater-like pits,

generally disconnected, now filled with asphalt, from which a variety of prehistoric flora and fauna have been excavated, including the trunks of trees, pine cones, acorns, mastodons, woolly mammoths, elephants, the ancient ox, giant sloth, camel, saber-toothed tiger, lion, horse, wolf, cave bear, and numerous species of vultures and

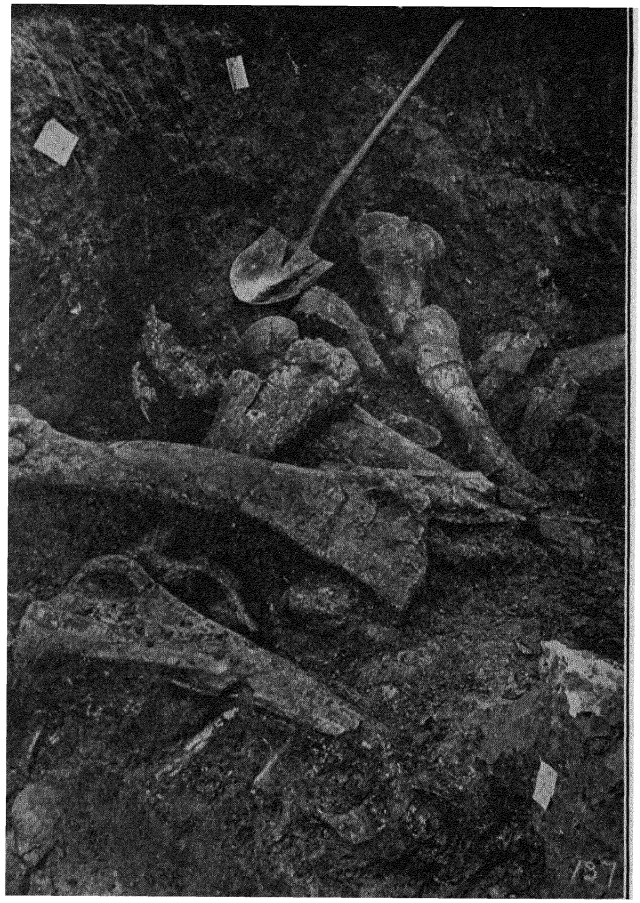


Courtesy Los Angeles Museum.

FIG. 2.—Cypress Tree Preserved by Asphalt for 25,000 Years, Rancho-la-Brea Asphalt Pits, California.

carnivorous birds, all now extinct. The theory has been advanced that the pits were originally formed by "blow-outs" of gas from an underlying oil deposit, forming surface craters of funnel-like shape, filled by an inflow of soft, sticky asphalt, which in time became quiescent, possibly crusting over, but deadly to any form of beast

that stepped into them. Once mired in the asphalt, the victim's struggles would only sink it deeper and attract a host of carnivores to the feast. These in turn would only contribute so many more victims to the insatiable greed of the trap. Then came carrion eaters, vultures, eagles and many others, which contributed avian



Courtesy Los Angeles Museum.

FIG. 3.—Bones of Pre-historic Animals Excavated from Rancho-la-Brea Asphalt Pits.

remains to the mass. An artist's conception of the "Death Trap" of Rancho-la-Brea is illustrated in Fig. 1. The skeletons have been preserved perfectly by the asphalt. They have been identified by archeologists as belonging to the pleistocene or glacial period, which according to authorities terminated about 25,000 years ago, or long before the advent of man.

Figure 2 shows the trunk of a pleistocene cypress found in one of the pits in an upright position, packed solidly about with bones of the aforementioned animals, and preserved in almost its original state by the asphalt which surrounded it. This represents one of the oldest specimens of wood in existence and bears mute evidence of the remarkable preservation properties of asphalt.

Figure 3 shows a mass of prehistoric elephant bones, as they were being excavated from the asphalt in an adjacent pit. In no other known mineral deposits are the bones of these huge beasts preserved as well as in the asphalt beds of Rancho-la-Brea. A smaller deposit of a similar character has been found near McKittrick, Cal.[2]

Use of Asphalt by the Sumerians (about 3800 to 2500 B.C.).[3] The earliest recorded use of asphalt by the human race was by the pre-Babylonian inhabitants of the Euphrates Valley in southeastern Mesopotamia, the present Iraq, formerly called Sumer and Accad (Akkad), and later Babylonia. In this region, between the river Nile in Egypt and the Indus river in India, there occur various deposits of asphalt as illustrated in Fig. 4. The following historical data have been recorded:

King Sargon of Accad (*about* 3800 *B.C.*). A legend has come down through the ages that King Sargon, while an infant, was placed in a reed basket coated with asphalt, by his mother Itti-Bel, a Priestess, and set adrift on the waters of the Euphrates river during one of its frequent overflows.[4] This corresponds closely with the tale of Moses, later appearing in the Bible.

Manishtusu, King of Kish (*about* 3600 *B.C.*). The bust of this early Sumerian ruler was found in the course of excavations at Susa, in Persia, whence it is supposed to have been carried by an Elamite conqueror in the twelfth century B.C.[5] In describing this statue, M. J. de Morgan states; "the eyes, composed of white limestone, once ornamented with black pupils now fallen off, are held in their orbits with the aid of *bitumen*; the face appears rough; the beard and hair are of conventional design; as regards the inscription, it is engraved in lineal cuneiform characters of the most ancient style." The original is at the Louvre and is illustrated in Fig. 5.

Ornaments (*about* 3500 *B.C.*). Fragments of a ring composed of asphalt have been unearthed above the flood layer of the Euphrates at the site of the prehistoric city of Ur in southern Baby-

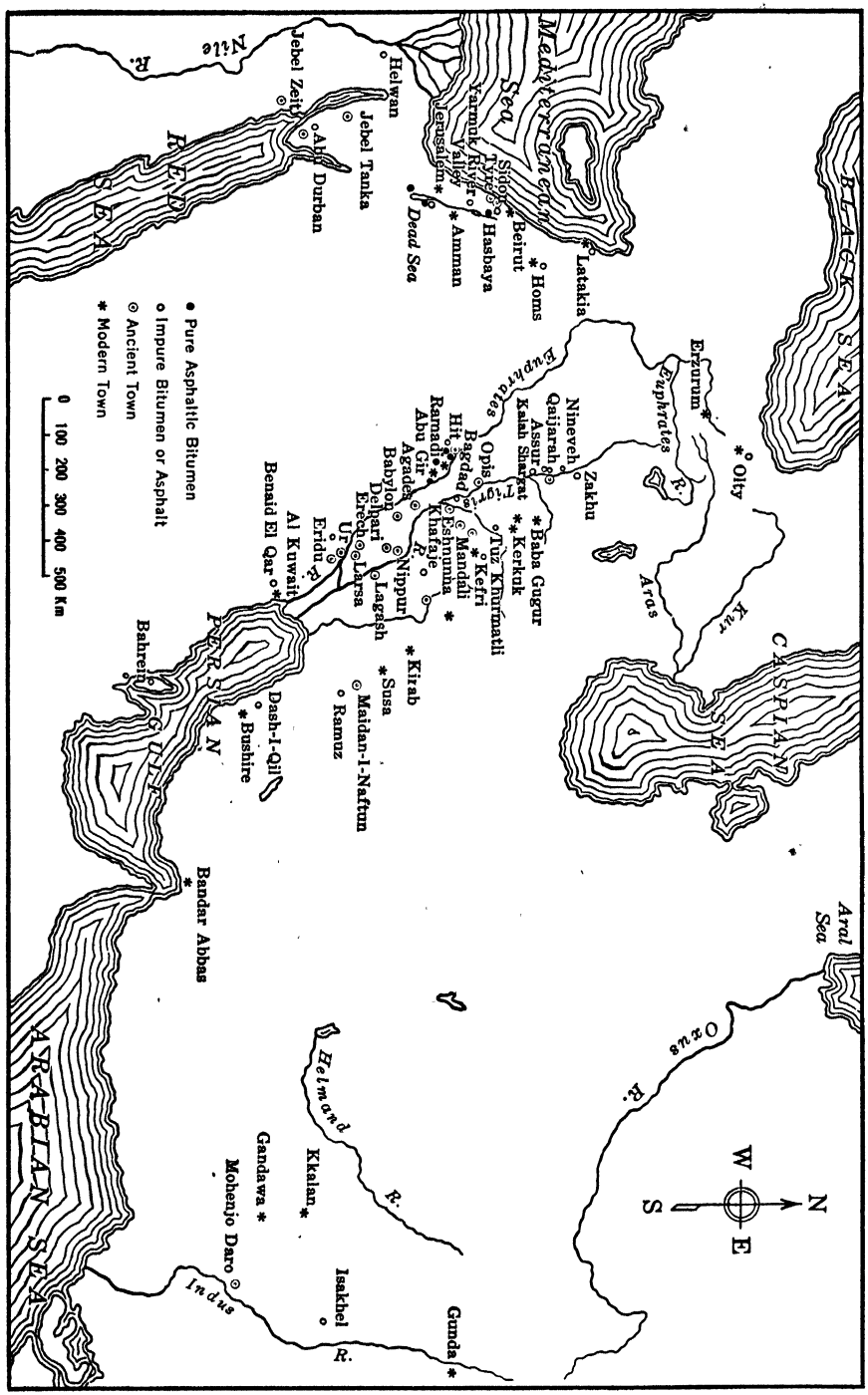


FIG. 4.—Asphalt Deposits between Nile and Indus Rivers.

lonia, ascribed to the Sumerians of about 3500 B.C.[6] Upon analysis it was found to consist of the following:

Soluble in pyridine	63.7 per cent
Ash recovered from pyridine extract	3.8 per cent
Ash not removed by pyridine	27.4 per cent
CO_2 equivalent to CaO in ash	1.9 per cent
Organic matter (by difference)	3.2 per cent
Total	100.0 per cent

The mineral constituents were composed of 51 per cent silica (sand, diatoms, foraminifera and cells) and 34 per cent $CaSO_4$.



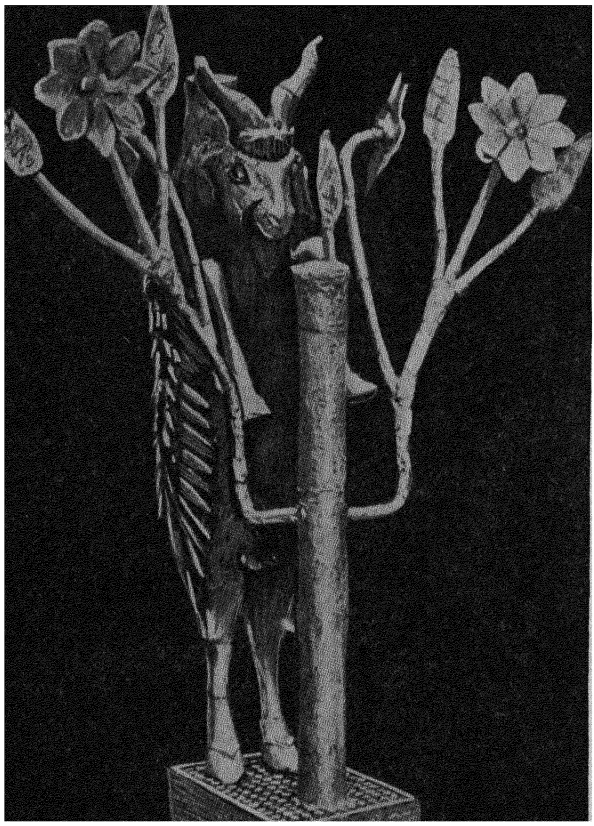
From "The Civilization of Babylonia and Assyria," J. B. Lippincott Co.

FIG. 5.—Bust of Manishtusu, King of Kish (3600 B.C.) with Eyes Set in Asphalt.

An interesting ornament has been excavated from one of the graves of a Sumerian King at Ur [7] consisting of a statue of a ram

dating back to 3500-3100 B.C. as illustrated in Fig. 6. The head and legs of the ram, also the tree-trunk are carved out of wood over which gold foil has been cemented by means of asphalt. The back and flanks of the ram are coated with asphalt in which hair has been embedded.

At Tell-Asmar in Eshnunna (3200 *to* 2900 *B.C.*). Excavations



(From F. A. Brockman, Leipzig. Courtesy of New York Public Library)

FIG. 6.—Sumerian Statue of a Ram (3500-3100 B.C.)

in 1931 at Tell-Asmar, 50 miles northeast of Baghdad, on the eastern bank of Diyala river (which joins the Tigris river directly south of Baghdad) have revealed the use of asphaltic mastic by the Sumerians for building purposes.[8] Similarly, at Khafaje, in the same vicinity, excavations have uncovered floors composed of a layer of asphalt mastic 3 to 6 cms. thick, likewise clay bricks bonded together with asphalt mastic. This mastic has been identified as

asphalt (probably originating in the vicinity of Hit), mineral filler (loam, limestone and marl) and vegetable fibers (straw). This

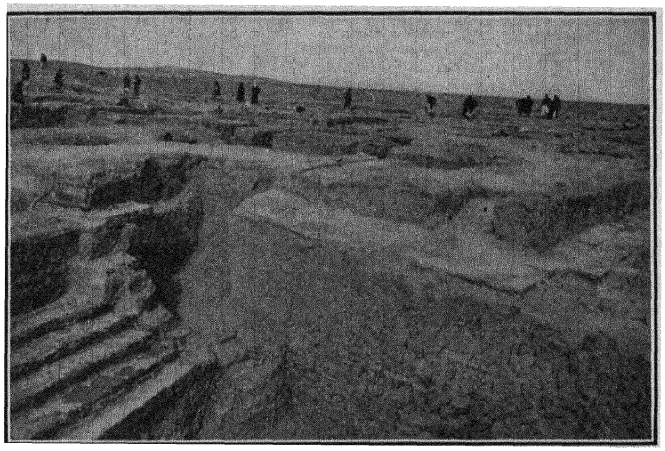


FIG. 7.—Babylonian Pavements Excavated at Tell-Asmar.

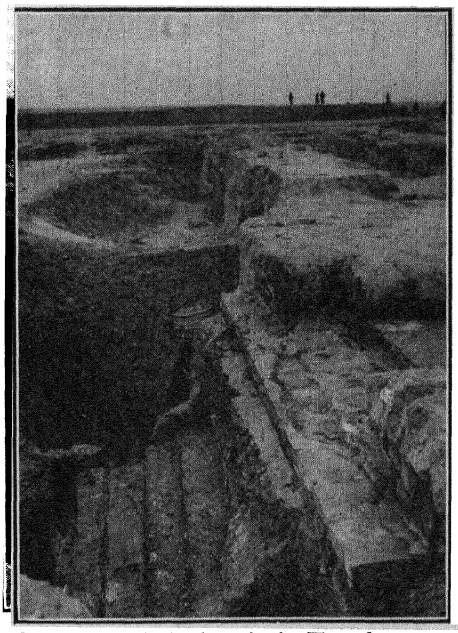


FIG. 8.—Babylonian Stair Treads at Tell-Asmar.

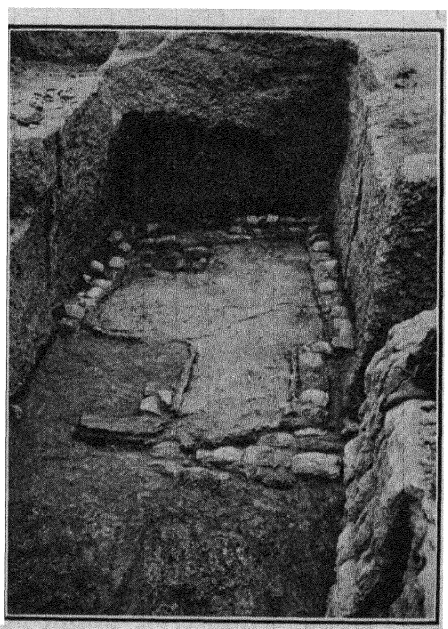
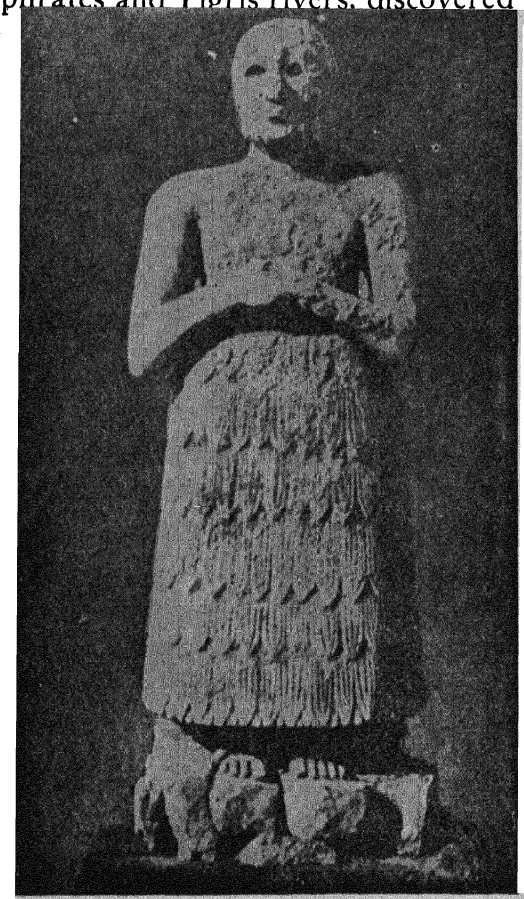


FIG. 9.—Babylonian Baths Waterproofed with Asphalt-Mastic at Tell-Asmar.

composition was used for bonding bricks in the construction of buildings and pavements (Fig. 7); for protecting exterior masonry surfaces; for troweling over the surface of interior floors and

stair treads (Fig. 8); and for waterproofing baths (Fig. 9), drains, etc.[9]

Lugal-daudu, King of Adab (*about* 3000 *B.C.*). In 1903–4, Dr. E. J. Banks, while excavating at Adab (known also as Bismaya, between the Euphrates and Tigris rivers, discovered a marble statue



From "The Civilization of Babylonia and Assyria," J. B. Lippincott Co.

FIG. 10.—Statue of Lugal-daudu, King of Adab (3000 B.C.) Showing Eyesockets Lined with Asphalt.

of Lugal-daudu, King of Adab (Fig. 10), one of the early Sumerian rulers, who lived about 3000 B.C.[10] An inscription reveals the name of the city of Adab. The eyesockets are hollow, and still show the presence of asphalt, indicating that they were once inlaid with some substance, probably ivory or mother-of-pearl. The statue is now on exhibition at the Ottoman Museum in Istanbul.[11]

Another statue (Fig. 11) originating about the same time (3000 B.C.), known as the "Human-Headed Bull," is composed of black steatite, inlaid with small yellow shells imitating streaks, and held in place with asphalt. Many of the shells are intact, gripped firmly by the asphalt throughout fifty centuries of time and exposure, thus furnishing evidence of its remarkable adhesiveness and durability. This statue is now at the Louvre, Paris.[12]



From "The Civilization of Babylonia and Assyria," J. B. Lippincott Co.

FIG. 11.—Human-Headed Bull (3000 B.C.) with Shells Inlaid in Asphalt.

Entemena of Shirpula (*about* 2850 *B.C.*). An interesting specimen of Sumerian art was excavated at Lagash, near the mouth of the Euphrates, consisting of a sculptured votive offering dating back to Entemena, ruler or so-called "Patesi" of Shirpula (2850 B.C.). This bears as an inscription, the heraldic device of Lagash, by means of which we are enabled to fix its date and origin. The tablet

is an artificial composition of clay and asphalt (Fig. 12). It is also on exhibition at the Louvre.[13]

Ur-Ninā, King of Lagash (*about* 2800 *B.C.*). In the city of Kish (Persia) there has been excavated the palace of King Ur-Ninā, the foundations of which consist of plano-convex bricks cemented together with asphalt mortar. Similarly, in the ancient city of Nippur (about 60 miles south of Baghdad) excavations show Sumerian structures dating from this same period, composed of natural



From "The Civilization of Babylonia and Assyria," J. B. Lippincott Co.

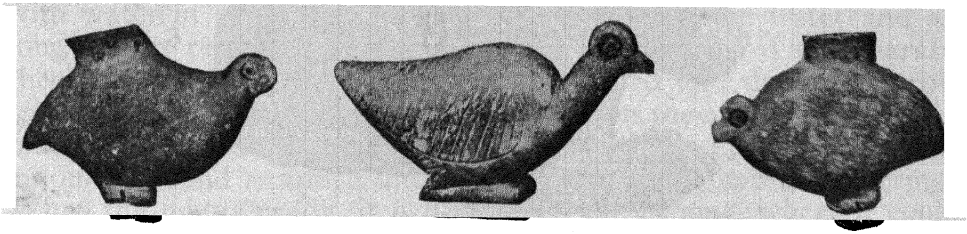
FIG. 12.—Heraldic Device of Lagash (2850 B.C.) Cast in Asphalt.

stones joined together with asphalt mortar, including the "ziggurat" of Enlil.

Ornaments and Sculptured Objects (2800 *to* 2500 *B.C.*). A number of specimens of sculpture involving the use of asphalt were excavated at Susa in the province of Susiana, by M. J. de Morgan's expedition of Paris.[14] These are in an excellent state of preservation, and by the inscriptions and characteristic orna-

mentation are supposed to have originated between 2800 and 2500 B.C.

Figure 13 shows various small animals carved of alabaster having the eyes cemented in place with asphalt; Fig. 14, two decorated vases composed wholly of asphalt; and Fig. 15, a sculpture



From "Mémoires de la Délégation en Perse," by Edm. Pottier.

FIG. 13.—Sumerian Sculpture with Eyes Set in Asphalt.

of an animal in primitive form, hewn from a mass of asphalt. The French chemist, Henri Le Chatelier, analyzed some of the asphalt, and found it to consist of the following:[15]

Moisture, 2.8 per cent; asphalt, 24.4 per cent; wax, 1.6 per cent; mineral matter, 71.2 per cent. The mineral matter was composed of: calcium carbonate, 45.2 per cent; calcium sulfate, 3.5 per cent; calcium phosphate, 0.8 per cent; iron, aluminium and silicon oxides, 21.7 per cent.



From "Mémoires de la Délégation en Perse," by Edm. Pottier.

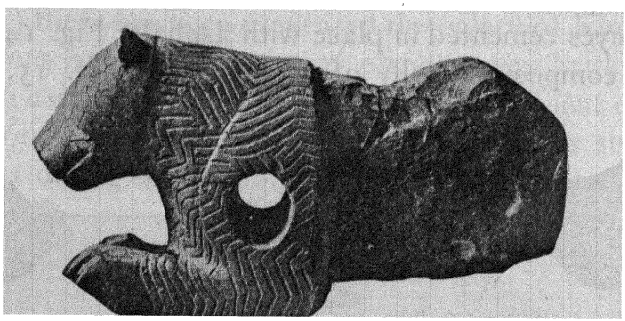
FIG. 14.—Persian Vases Hewn from Blocks of Asphalt.

This is conclusive proof that the asphalt is a natural product composed of 25 per cent asphalt and 75 per cent mineral matter, similar to the material obtained in the locality at the present day.

Small statues of Sumerian origin (about 2500 B.C.) have been unearthed in Mesopotamia, composed of white clay with wigs of asphalt and blobs of red paint on the cheeks.[16]

Gudea of Lagash (about 2700 B.C.). Another relic, known as the "Libation Vase" (Fig. 16) is composed of green steatite, carved

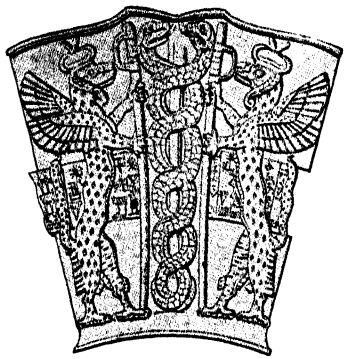
in the form of strange mythical monsters, the effect of which is heightened by incrusted little shells set in asphalt, to represent the



From "Mémoires de la Délégation en Perse," by Edm. Pottier.

FIG. 15.—Primitive Animal Carved from Asphalt.

scaly backs of winged serpents. The serpent was supposed to represent the emblem of the god Ningishzida, to whom the accompanying inscription shows the vase to be dedicated by Gudea, ruler of Patesi of Lagash. This is considered one of the best specimens of Sumerian sculpture, and represents the height of Sumerian art. It is also at the Louvre.[17]



From "The Civilization of Babylonia and Assyria," J. B. Lippincott Co.

FIG. 16.—Libation Vase Dedicated to Gudea, Ruler of Lagash (2700 B.C.), Showing Shells Set In Asphalt.

Another inscription credited to Gudea states:[18]

"Asphalt from the Magda mountains in Elam was transported to his City of Lagash."

Tablets of Gilgamish (*about* 2500 *B.C.*). In the epic of Gilgamish as revealed in the twelve inscribed stone tablets collected by Assur-bani-pal, king of Assyria (668-626 B.C.), we find reference to the use of asphalt for building purposes. These tablets date back to about 2500 B.C. and constitute one of the most important pre-Babylonian literary records. In the eleventh tablet, Ut-Napishtim relates the well-known story of the Babylonian flood, stating that he "smeared the inside of a boat with six sar (measures) of kupru (asphalt) and the outside with three sar."[19]

The Persian writer al-Kazwînî [20] gives the following interesting information relative to the collection and treatment of asphalt found in ancient Persia:

"There are two kinds of native asphalt. First the kind that oozes from certain mountains; second we have the kind that appears with water in certain pools. When boiled with the water and as long as they remain together, the asphalt is soft; but if we separate them, the asphalt hardens and becomes hard and dry. It is collected by means of matting and deposited on the shore. Then it is placed in a kettle under which a fire has been lit, and a certain amount of sand is added and a mix prepared by constant stirring. When the mix is ready, it is poured on the ground, where it cools and hardens."

In ancient texts we are informed that asphalt mastic was always sold by volume, namely, by the so-called "gur," the measure used for grain, beer, etc., as is evidenced by the very name Ur. Crude asphalt, on the other hand, was always sold by weight, by the "mina" or "shekel," as were metals and other solid commodities.

Bur-Sin, King of Ur (*about* 2500 *B.C.*) An ancient Sumerian chapel erected to the Moon God "Nin-Gal" has recently been excavated, in which the floors are composed of burnt bricks embedded in asphalt mastic which still shows impressions of reeds with which it must originally have been mixed, adorned with stone tablets, each of which bears the following inscription:

"Bur-Sin, King of Ur, of Sumer and Accad, King of the four portions of the World, has constructed this for his Master Nin-Gal"

Use of Asphalt by Pre-historic Races in India (about 3000 B.C.). At excavations conducted in 1923 at Mohenjo-Daro, Harappa and Nal, in the Indus valley, northwestern India, evidences of an advanced form of civilization have been revealed.[21] An asphalt mastic, composed of a mixture of asphalt, clay, gypsum and organic matter, was introduced between two brick walls in a layer about 1-in. thick for waterproofing purposes. A bathing pool measuring 39 by 23 by 8 feet in depth has been unearthed, located in front of an ancient temple, probably for ritualistic cleansing purposes, containing a layer of the mastic on the outside of its walls and beneath its floor,[22] as illustrated in Figs. 17 and 18.

The mastic was found to have the following composition:

Organic constituents	28.3 per cent
Mineral constituents	71.7 per cent
Total	100.0 per cent

The organic constituents were 29.3% soluble in carbon disulfide and 85.5% in pyridine, and contained 7.1% sulfur. The mineral constituents contained: SiO_2 and insoluble: 55.1%; Fe_2O_3 and Al_2O_3: 11.2%; $CaSO_4$: 13.7%; CaO: 4.8%; $MgCO_3$: 4.2%;

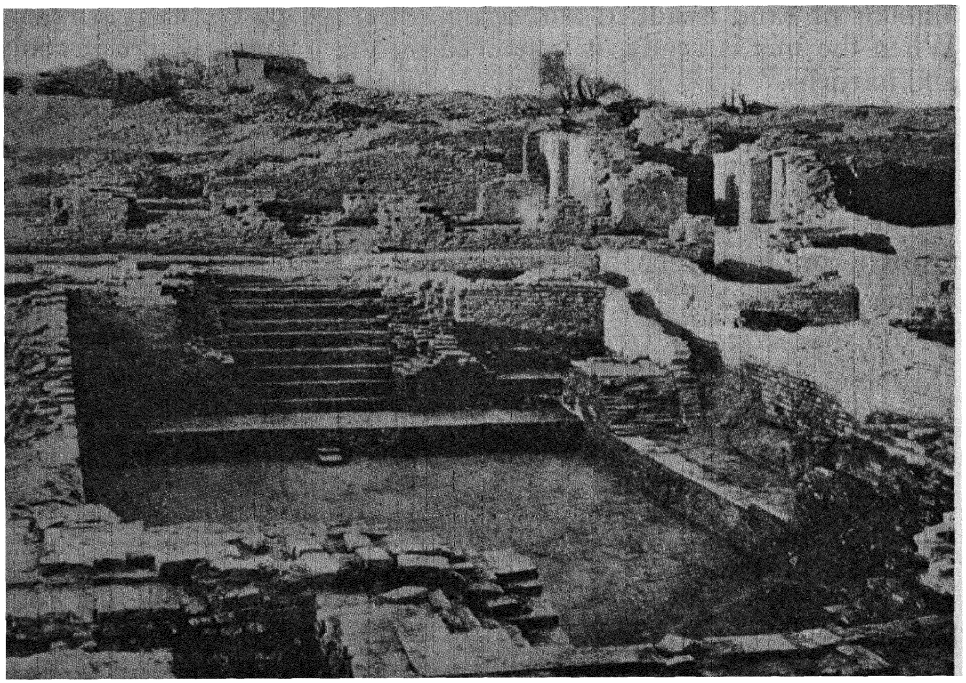


Photo Archeol. Survey of India

FIG. 17.—Water Tank in Front of a Temple Excavated at Mohenjo Daro (Indus Valley).

vanadium and nickel: traces. This mastic was also used for mosaic and inlaid work, as an adhesive for the application of ornaments to statues, and as a protective coating for woodwork. Seepages of liquid asphalt, as well as rock asphalt deposits exist today in India, on the Basti river, near Isakhel (Kashmir) and in the Sierras in Hazara District.

Use of Asphalt by the Early Egyptians (2500 to 1500 B.C.).[23] The ancient Egyptians were the first to adopt the practice of em-

balming their dead rulers and wrapping the bodies in cloth which was coated with liquid, or melted, solid waterproofing substances, including balsams, oleo-resins, gum-resins, true resins, wood-tar pitch (obtained during the process of charcoal making), and frequently with natural asphalt, although Alfred Lucas, formerly director of the Chemical Department of Egypt, has recently contended that he has been unable to detect asphalt in the black substance covering embalmed mummies, and concludes that resin

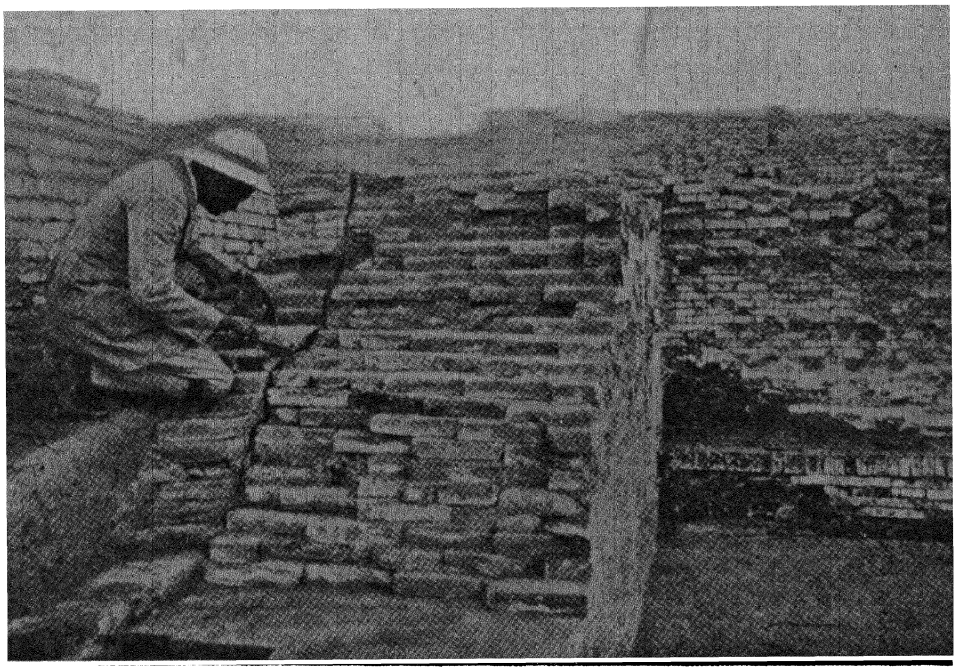


Photo Archeol. Survey of India

FIG. 18.—Wall of Water Tank at Mohenjo Daro, Showing the Bituminous Layer.

was employed for this purpose. Strabo [24] informs us that "the Egyptians used the Dead Sea asphalt for embalming their dead," and this practice is also alluded to by Diodorus Siculus, Pliny the Elder, Dioscorides, and other writers (loc. cit.). The preserved remains are known as "mummies" (Fig. 19).

Before 1000 or 900 B.C. asphalt was rarely used in mummification, except to coat the cloth wrappings and thereby protect the body from the elements. After the viscera had been removed, the cavities were filled with a mixture of resins and spices, the corpse

immersed in a bath of potash or soda, dried, and finally wrapped From 500 to about 40 B.C. (i.e. in Roman times), asphalt was generally used both to fill the corpse cavities, as well as to coat the cloth wrappings.[25] The word "mûmîa" first made its appearance in Arabian and Byzantine literature about 1000 A.D.,[26] signifying "bitumen." In Persian it acquired the meaning "paraffin wax," and in Syriac alluded to "substances used for mummification."

The product "mûmîa" was used in prescriptions, as early as the 12th century, by the famous Arabian physician Al Magor, for the

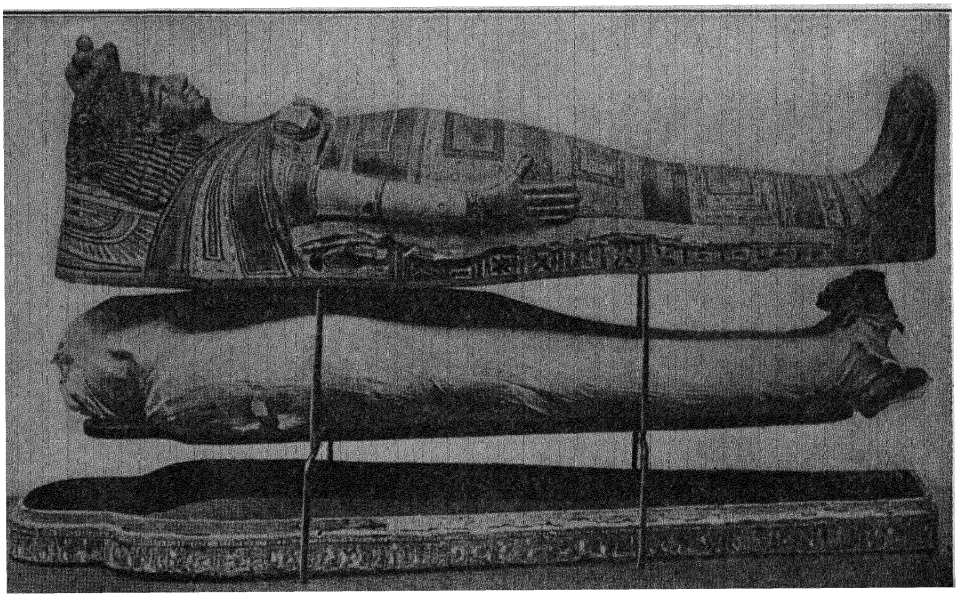


FIG. 19.—Mummy Preserved with Asphalt.

treatment of contusions and wounds. Its production soon became a special industry in the hands of the Alexandrian Jews. As the supply of mummies was of course limited, other expedients came into vogue. The corpses of slaves or criminals were filled with asphalt, swathed and artificially aged in the sun. This deception continued for several centuries until in 1564 A.D., it was exposed after a journey into Egypt by the French physician, Guy de la Fontaine, and as a result, this trade became extinct in the 17th century.

The earliest mummies in existence concerning which we have authentic data, date back to the Sixth Egyptian Dynasty (about 2500 B.C.). The earliest specimens include the mummy of Seker-

em-sa-f, unearthed at Sakkarah in 1881, and exhibited at Gîza, near Cairo,[27] and the mummy of King Merenrē, now at the Boulak Museum, Cairo.[28]

Tut-ankh-amen (or Tutenkhamûn), the boy Pharaoh, who ruled Egypt about 2000 B.C. and whose treasure-filled tomb was discovered by Lord Carnarvon of England in 1923, contained two statues of the Pharaoh, a black box, a figure of a swan, a couch, an ancient chariot, and numerous other wooden objects preserved by impregnation with asphalt.

Use of Asphalt in Biblical Times (2500 to 1500 B.C.).[29] Some contend that Noah used asphalt in the construction of the Ark (Genesis VI, 4). The Biblical text reads, that it was treated with "pitch" within and without: "bituminabis eam bituminae" (Vulgate). There is little doubt but that asphalt was used for this purpose, since it is well known that canoes and dugouts in the early days were made water-tight in the same way. In fact, a number of primitive tribes to-day adopt the same procedure. If asphalt was actually used by Noah, the date would be fixed at approximately 2500 B.C., which is usually assigned to the Deluge.

We find numerous other references in the scriptures to substances corresponding to what we now know to be asphalt. The Book of Genesis (XI, 3) in describing the building of the Tower of Babel (about 2000 B.C.) states ". . . and they had brick for stone, and *slime* had they for mortar." There seems to be no question but that the so-called "slime" alludes to asphalt, since the word translated as "slime" in the English version (1568), occurs as ἄσφαλτος in the Septuagint, and as "bitumen" in the Vulgate.

In the Septuagint, or Greek version of the Bible, this word is translated as "asphaltos," and in the Vulgate or Latin version, as "bitumen." In the Bishop's Bible of 1568 and in subsequent translations into English, the word is given as "slime." In the Douay translation of 1600, it is "bitume," while in Luther's German version, it appears as "Thon," the German word for clay.

Similarly, in Genesis (XIV, 10) we are informed that the Vale of Siddim "was full of *slime*pits," referring no doubt to exudations of liquid asphalt. Moreover, it is pointed out by certain authorities that the area described as the Vale of Siddim corresponds to our present Dead Sea, from which asphalt is still obtained.

Again we are told (Exodus II, 3) that in constructing the basket of bulrushes in which Moses was placed, it was daubed "with *slime* and with *pitch*." This took place about 1500 B.C.[30] As was pointed out previously, this constituted an early method of constructing boats. Even at the present time, boats known as "guffas" as illustrated in Fig. 20 are constructed of woven reeds caulked with as-

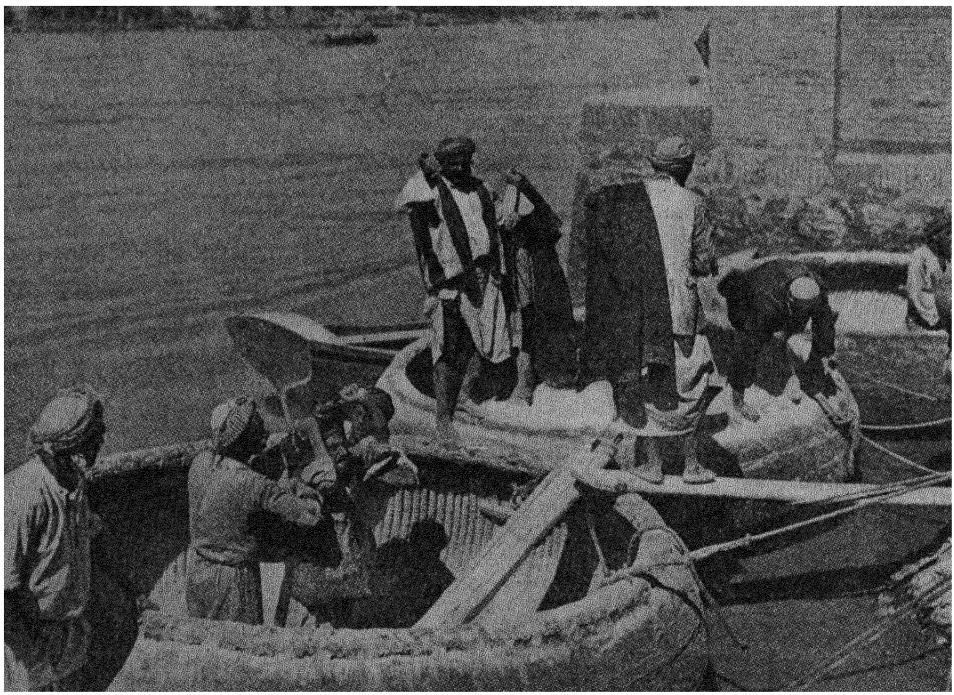


FIG. 20.—The Guffa, or Coracle, Used for Centuries in Parts of the Near East.

phalt,[31] and are used to ferry passengers and merchandise across the Tigris river at Baghdad.

While excavating at the ancient Biblical city of Jericho in Palestine,[32] brick walls were found in which asphalt mastic was used as mortar, dating back to about 2500–2100 B.C.

It is also contended that the glance pitch deposit known as "Sûk-El-Chan," on the western slope of Mount Hermon in the upper Jordan valley, near Hasbaya (loc. cit.) has been worked since about 1600 B.C.

Use of Asphalt by the Babylonians (2500 to 538 B.C.). Babylonia was the name given to the plain of the Tigris and Euphrates

rivers, now forming the modern province of Irak. The Babylonians were well versed in the art of building, and each monarch commemorated his reign and perpetuated his name by constructing some vast engineering work. Certain kings built roadways, others built retaining walls to impound the waters of the Euphrates, and still others mighty battlements and palaces. Such facts were indelibly recorded by inscriptions on the bricks used for the purpose, many of which are still in existence. The bricks were of the so-called "plano-convex" type, being formed by hand, with one side flat and the other face convex and then kiln burnt. Bitumen was used as mortar from very early times. Sand, gravel or clay were employed in preparing these mastics. Due to the porous nature of the bricks, the asphalt was partially absorbed, resulting in a very strong bond. At first, the spaces between the bricks was filled with a 3 to 6 cm. layer of mastic, but in later periods there was a tendency to diminish the space.

Asphalt-coated tree trunks, e.g. poplar, were often used to reinforce wall corners and joints, as for instance in the temple tower of Ninmach in Babylon. In vaults or arches a mastic-loam composition was used as mortar for the bricks, and the keystone was usually dipped in asphalt before being set in place. Reuther reports that bituminous paint was applied to the outer walls of buildings in Babylon over a loam plaster, first primed with a gypsum distemper. Asphalt was also used to waterproof brick basins at Ur and Erech.[33]

King Khammurabi (*about* 2200 *B.C.*). The use of bituminous mortar was said to have been introduced in the city of Babylon by King Khammurabi (Amraphel of the Bible). Robert Koldewey reports that when excavating in Babylon, he found "it was exceedingly difficult to separate the brick courses from each other." [34] This was also confirmed by A. H. Layard, who states that "Bricks bonded with asphalt have remained immovably in place for thousands of years." [35]

Queen Semiramis (*about* 700 *B.C.*) Queen Semiramis is stated to have built a tunnel under the Euphrates at Babylon, about 1000 meters long, of burnt bricks coated with asphalt as mortar.[36] The asphalt thus functioned as a waterproofing agent, very much in the same manner as it is used to-day.

King Nabopolassar (625 *to* 604 *B.C.*). Nabopolassar is cred-

ited with having built a palace on a platform consisting of ten courses of burnt bricks set with an asphalt mortar and surfaced with a layer of asphalt mastic. One of his inscriptions states:

"I made a Nabalu and laid its foundations against the bosom of the underworld, on the surface of the water, in bitumen and brick. I raised its roof and connected the terrace with the palace. With bitumen and brick, I made it tall like unto wooded mountains."

According to his son Nebuchadnezzer, he is given the credit to have laid the first asphalt block pavement in Babylon.

King Nebuchadnezzar (604 *to* 561 *B.C.*). Of all the Babylonian rulers, Nebuchadnezzar was the most progressive, and is stated to have reconstructed the entire city. The bricks bore inscriptions relating to his work, and several refer specifically to the use of asphalt. One found in the so-called "Procession Street" (Aibursha bu) which led from his palace to the North wall, reads as follows:[87]

"Nebuchadnezzar, King of Babylon, he who made Esaglia and Ezida glorious, son of Nabopolassar, King of Babylon. The streets of Babylon, the Procession Street of Nabû and Marduk, my lords, which Nabopolassar, King of Babylon, the father who begot me, had made a *road glistening with asphalt* and burnt bricks; I, the wise suppliant who fears their lordships, placed above the *bitumen* and burnt bricks, a mighty superstructure of shining dust, made them strong within with *bitumen and burnt bricks* as a high-lying road. Nabû and Marduk, when you traverse these streets in joy, may benefits for me rest upon your lips; life for distant days, and well-being for the body. Before you I will advance upon them. May I attain eternal age!"

This street was constructed with stone slabs brought from distant parts, set in a bituminous mortar, the interstices being very narrow at the surface and widening towards the base of the stones. The foundation consisted of three or more courses of bricks joined together with bituminous mortar.

This would seem to be the forerunner of the present-day pavement composed of stone blocks set in asphalt. It seems strange that the art should have become lost to mankind, only to be rediscovered in the nineteenth century A.D.

The most comprehensive relic left by Nebuchadnezzar is known

as the "Large Inscribed Stone Tablet" (sometimes referred to as the "East India House Inscription"), which contains a detailed account of his building activities. A translation by Fr. Delitsch [38] reads in part as follows (Column 7, lines 34 *et seq.*):

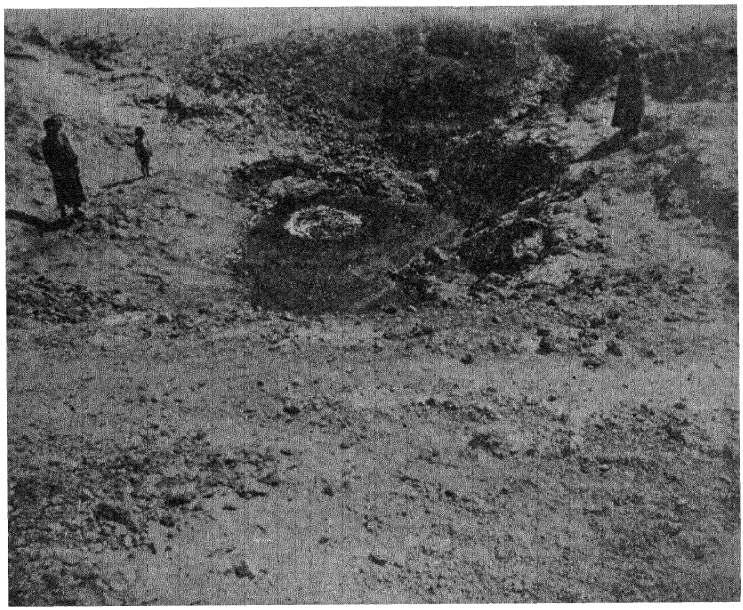
"In Babil, my favorite city that I love, was the palace, the house, the marvel of mankind, the center of the land, the dwelling of majesty, upon the Babil place in Babil, from Imgur-Bel to the eastern canal Libil-Higalla; from the bank of the Euphrates to Aiburshabu, which Nabopolassar, King of Babylon, my father, my begetter, built of *crude bricks,* and dwelt in it. In consequence of high waters, its foundations had become weak, and owing to the filling up of the streets of Babil, the gateway of that palace had become too low. I tore down its walls of *dried brick*, and laid its corner-stone bare, and reached the depth of the waters. Facing the water, I laid its foundation firmly, and raised it mountain high with *bitumen and burnt brick.* Mighty cedars, I caused to be laid down at length for its roofing. . . . For protection, I built two massive walls of *asphalt* and brick, 490 ells beyond Nimitti-Bel. Between them I erected a structure of bricks on which I built my kingly dwelling of *asphalt and bricks.* This I surrounded with a massive wall of *asphalt and burnt bricks*, and made upon it a lofty foundation for my royal dwelling of *asphalt and burnt bricks.*"

It thus appears that Nebuchadnezzar profited by the experience of his father, and instead of building a retaining wall of dried clay bricks, which had failed to hold back the Euphrates due to its lack of waterproof properties, he resorted to the use of burnt bricks and *asphalt*, and apparently with satisfactory results.

Robert Koldewey's investigations indicate that the method of constructing walls in Babylonia and Nineveh consisted in laying in rotation, first a course of bricks, then a layer of asphalt, then a layer of clay and then another course of bricks.[39] The joints in each course were composed of asphalt and clay. In every fifth course, the clay was replaced by a matting of reeds. This matting is now entirely rotted and gone, but its impression is clearly recognizable in the asphalt. An attempt to separate the courses to prevent adhesion is thus apparent, but the reason is not obvious. Only in one locality (Temple of Borsippa) does it appear that asphalt has been used in direct contact with the bricks, where they still hold together in a firm mass.

It is probable that the asphalt used by the Babylonians was derived from springs similar to the ones still found in Mesopotamia, of which Fig. 21 is a typical example of an asphalt spring at Hit. Deposits of pure asphalt are found to-day at various localities in this region, including Ain Mamurah, Ain-el-Maraj, Al-Kuwait (Arabia), Bushire (Iran), and Bundar Abbas (Iran).

Figure 22 shows the present appearance of the brick floor of Nebuchadnezzar's Throne Hall, Babylon, looking towards the



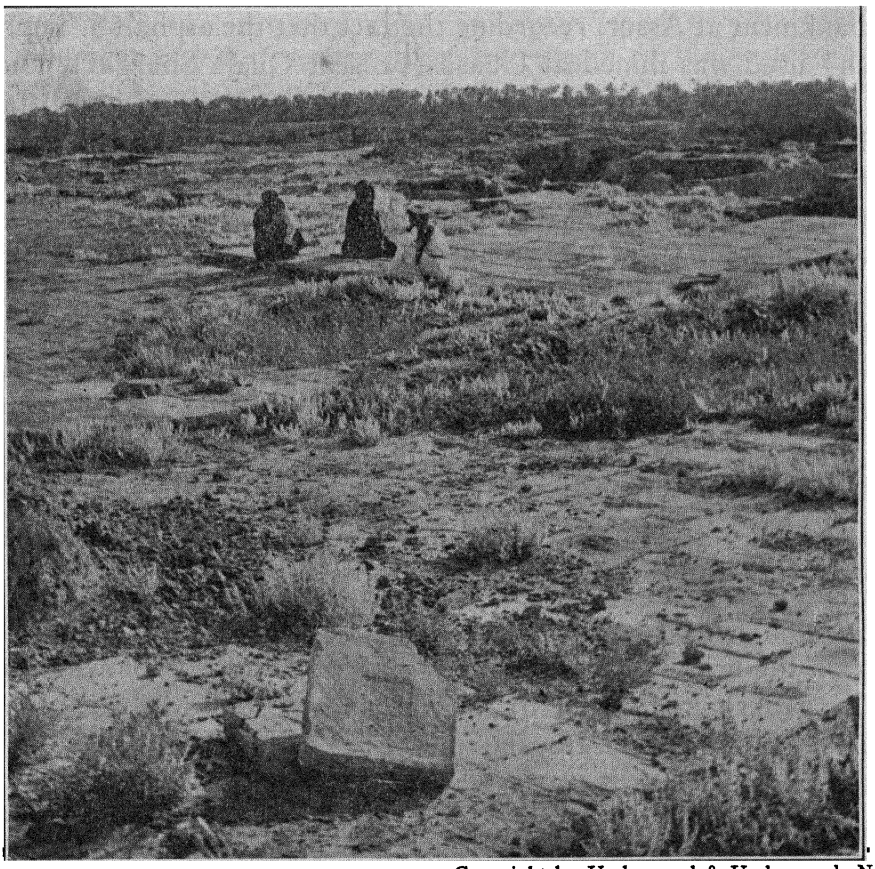
From "Light on the Old Testament," by Prof. A. T. Clay.

FIG. 21.—Asphalt Spring in Mesopotamia.

Euphrates. The burnt bricks bearing the name of Nebuchadnezzar (of which one is shown in the foreground) were laid in asphalt, and are still so firmly jointed together to-day, that it is impossible to part them without destroying their integrity.[40]

King Nebuchadnezzar also built a bridge across the Euphrates near Babylon, 370 feet long. The piers were constructed of burnt bricks embedded in asphalt mastic, the bases of which were protected with a superficial coating of asphalt. He also constructed large sewers ("cloacae") for draining the city of Babylon, lined with blocks composed of a mixture of asphalt, loam and gravel.

The use of bituminous mortar was abandoned in Babylon towards the end of Nebuchadnezzar's reign, in favor of lime mortar to which varying amounts of asphalt were added. After the fall of Babylon (538 B.C.) even the addition of asphalt was discontinued. In the ensuing Persian period (538 to 300 B.C.), the



Copyright by Underwood & Underwood, N. Y.

FIG. 22.—Floor of Nebuchadnezzar's Temple as it Appears To-day, Showing Blocks Joined by Means of Asphalt.

lime was replaced in turn with clay as mortar, and this resulted in a retrogression in the building art.

Use of Asphalt by the Assyrians (1400 to 607 B.C.).[41] The name Assyria was derived from the city of Assur (now Kalah Shargat) situated on the right bank of the Tigris, midway between the Greater and Lesser Zab rivers. Assur was finally supplanted

by Nineveh as capital (Nebi Yanus and Kuyunjik) some 60 miles farther north.

King Adad-Nirari I (*about* 1300 *B.C.*). This monarch is stated to have built an embankment 1 mile long along the banks of the Tigris, in which the stones were joined together with an asphaltic mortar. W. Andrae reports that tablets have been found in the embankment at Assur, recording the fact that the asphalt ("kupru": pitch) used was mined at Ubasê (present Quala Shargat), and in reference to the mortar states that "After 3300 years, it still faithfully fulfils its purpose." [42] Andrae likewise refers to an ancient temple mound excavated at the former site of Erech, dating back to this same period, which was constructed of courses of dried clay bricks bound together with a mortar composed of asphalt mixed with clay. It is interesting to note in passing, that in the ancient Assyrian moral code we find reference to asphalt, which was prescribed to be poured onto the heads of delinquents in a molten state.

King Tukulti-Ninurta II (890 *to* 884 *B.C.*). This King, in his annals makes reference to the fact: [43]

"In front of Hit, by the bitumen springs—the place of Ušmeta Stones where the gods speak—I spent the night."

King Sargon (722 *to* 705 *B.C.*). The following inscription occurs on the bricks of the so-called "Sargon Wall" of Babylon, built by King Sargon, founder of the last and most illustrious Assyrian dynasty, as it has been translated by Fr. Delitsch: [44]

"To Marduk, the Great Lord, the divine Creator, who inhabits Esagila, the Lord of Babil, his lord Sargon, the mighty king, King of the land of Assur, King of all, governor of Babil, King of Sumer and Akkad, the nourisher of Esagila and Ezida. To build Imurg-Bel was his desire; he caused burnt brick of pure Kirû (?) to be struck, built a kâr (?) with *tar* and *asphalt* on the side of the Ishtar Gate to the bank of the Euphrates in the depth of the water, and founded Imgur-Bel and Nimitti-Bel mountain high, firm upon it. This work may Marduk, the great Lord, graciously behold, and grant Sargon, the prince who cherishes him, life! Like the foundation stone of the Sacred City, may the years of his reign endure."

"Imgur-Bel" was the name given to the inner wall of Babylon, and "Nimitti-Bel" to the outer.

King Sennacherib (704 *to* 682 *B.C.*). One of this King's inscriptions [45] informs us that he:

"Covered the bed of the diverted river Telbiti with rush matting at the bottom and quarried stone on top, cemented together with natural pitch (asphalt). I thus had a stretch of land 454 ells long and 289 ells wide, raised out of the water and changed into dry land" (Inscription No. 6).

The year 607 B.C. marked the destruction of Nineveh and the end of the Assyrian empire.

Use of Asphalt in Constructing Lake-Dwellings (about 1000 B.C.). In the bronze-age, dwellings were constructed on piles in lakes close to the shore the better to protect the inhabitants from the ravages of wild animals and attacks from marauders. Excavations have shown that the wooden piles were preserved from decay by coating with asphalt. Remains preserved in this manner have been found in Switzerland, in the lakes of Bourget, Geneva, Neuchâtel, Bienne, Zurich and Constance.[46]

References to Bituminous Substances by Greek and Roman Writers (500 B.C. to 817 A.D.)

Herodotus (484 *to* 425 *B.C.*). The Greek historian Herodotus, in his treatise "Historiarum" [47] refers to deposits of asphalt at Hit (the present town of Kirkuk in Mesopotamia) as follows:

"There is a city called Is (Hit), eight days' journey from Babylon, where a little river flows, also named Is, a tributary stream of the river Euphrates. From the source of this river many 'gouts' of asphalt rise with the water; and from thence the asphalt is brought for the walls of Babylon."

Many references occur in the earliest writings to Hit (Accadian "Id," Greek "Is"). A more recent though interesting description of the asphalt deposit at Hit is furnished by a British traveler George Rawlinson (1745),[48] who described his visit to that locality as follows:

"Having spent three days or better among the ruins of old Babylon, we came into a town called Hit, inhabited only by Arabians, but very ruinous. Near unto this town is a valley of pitch, very marvelous to behold, and a thing almost incredible, wherein are many springs throwing out abundantly a kind of black substance,

like unto tar and pitch, which serveth all the countries thereabouts to make staunch their barks and boats. Every one of which springs maketh a noise like a smith's forge in puffing out the matter, which never ceaseth night or day, and the noise is heard a mile off, swallowing all the weighty things that come upon it. The Moors call it the Mouth of Hell."

A translation of a tablet unearthed at Karnak[49] reveals the fact that part of the tribute paid Thothmes III (Tethmosis III), about 1500 B.C. from Mesopotamian cities consisted of 2040 minae (about 1000 kg.) of "zift" (Arabic for native asphalt), sent by the ruler of Is (Hit),[50] but the foregoing has been controverted.

Herodotus also states (VI, 119) that asphalt was obtained from wells near Ardericca (near the present town of Kirab) in the land of Cissia (now Persia):

"At Ardericca, 210 stadia from Susa, there is a well another 40 stadia away, which produces three different substances, since asphalt, salt and oil are drawn up from it. . . . It assumes three different forms: the asphalt and the salt immediately become solid, but the oil they collect, and the Persians call it Rhadinance; it is black and emits a strong odor."

At Elam, in the province of Susiana in Persia, asphalt is still collected in this crude manner.

Herodotus refers (IV, 195) to a deposit of pure, soft asphalt at Zacynthus (now Zante), on the Greek coast, as follows:

"I myself have seen the pitch drawn up out a pool of water in Zacynthus, and there are several pools there, the largest of which is 70 orgyoe (i.e. fathoms) long and broad, and 2 orgyoe in depth. Into this, they let down a pole with a myrtle branch fastened to its end, and then draw up the pitch adhering to the myrtle. It has the smell of asphalt, but in other respects is better than the pitch of Pieria (now Thessalonica). Then they pour it into a pit that has been dug near the pool; and when much is collected, they fill their vessels from the pit."

Herodotus was the first writer to describe the construction of small round boats woven together of reeds, like baskets, covered on the outside with hides, and waterproofed with a coating of natural asphalt, similar to the present-day "guffas" found in Mesopotamia (I, 194). He also described the use of bituminous mortar, com-

posed of asphalt and straw, for joining together stones and clay bricks in building masonry walls at Media.

Thucydides (471 *to* 401 *B.C.*). The Greek hsitorian Thucydides [51] refers to the use of petroleum for military purposes and describes a mixture of asphalt and sulfur, which was used to ignite fascines piled against the wooden walls in the sieges of Plataea and Delium.

Hippocrates (460 *to* 377 *B.C.*). This Greek philosopher and physician [52] refers to several of the asphalt deposits already mentioned.

Xenophon (430 *to* 357 *B.C.*). The Greek historian Xenophon [53] describes a wall built in Media, composed of burnt bricks cemented together with asphalt, similar to the method of construction used in Babylon.

Aristotle (384 *to* 322 *B.C.*). This Greek writer and philosopher [54] states:

"Inflammable oils rise in large quantities in the soil of Persia, and in smaller quantities in Sicily, where they often have the distinct odor of cedar resin. Thick, dark and viscous oils flow beside natural pitch and asphalt in Macedonia, Thrace and Illyria (Sélénitza in Albania) from the hot, often burning soil, smelling of sulfur and bitumen, and diffuse stinking, choking and sometimes deadly fumes."

Theophrastus (372 *to* 288 *B.C.*). This Greek writer [55] describes the production of wood tar, which he states was an established industry. He likewise gives a comprehensive account of several occurrences of asphalt, and refers to:

"An earth in Cilicia (present Turkey-in-Asia), which becomes viscous on heating."

Antigonus (*about* 311 *B.C.*). We are informed [56] that Antigonus of Macedonia in 311 B.C. sent Hieronymus of Cardia with an army to capture the asphalt works at the Dead Sea from the Nabataeans. This deposit seems to have been greatly prized. Later, Ptolemy II (309–246 B.C.) in turn, captured the Dead Sea asphalt works. It then passed on to the Seleucids, from whom Antony took it and presented it to Cleopatra. The latter leased it to Malchus, the Nabataean, in 36 B.C., who in 32–31 B.C. failed to

pay rent and was accordingly punished by Herod. The subsequent history of this most interesting deposit is lost in obscurity.

Hannibal (247 *to* 183 *B.C.*). The Carthaginian general Hannibal is said to have used asphalt in compounding the so-called Greek Fire ("Ignae Vestoe"), which was claimed to burn so fiercely, that even water would not extinguish it, and was used extensively by him in warfare.

Vergil (70 *to* 19 *B.C.*). This Roman poet [57] refers to the medicinal use of bitumen, applied externally as a cure for scabies.

Strabo (63 *B.C. to* 24 *A.D.*). The Greek geographer Strabo [58] refers in detail to the Dead Sea asphalt deposit. This was termed "Lacus Asphaltites" by the classical writers, and the asphalt derived therefrom, as "Bitumen Judaicum." Strabo states (XVI, 740):

"The Dead Sea is full of asphalt. It comes to the surface irregularly at the center, with noisy disturbance of the water, which appears as though it were boiling. The visible portion of the asphalt lumps is round and has the form of a hillock. Much sooty matter accompanies the gaseous emanation, which tarnishes copper, silver and all bright metals, and even causes gold to rust."

He adds further (XVI, 764):

"The asphalt remains on the surface of the water, owing to its salty nature. People go in rafts to the asphalt, hack it to pieces, and take as much of it away with them as they can."

He also offers the following interesting theory regarding the origin and formation of Dead Sea asphalt (XVI, 763):

"Asphalt is a lumpy earth, which has been liquefied by heat and again comes forth and spreads out, and eventually becomes converted into a dense, solid mass by the cold sea water, so that it must be worked with knives and axes. Undoubtedly, this occurs in the center of the lake, since the fire originates there and likewise the asphalt. The asphalt is generated irregularly, since the movement of the fire, the force of the wind, etc., have no marked regularity."

Strabo has the following to offer regarding Babylonian asphalt (I, 16):

"Poseidonius (died 51 B.C.) declares that there are wells of black and also white naphtha in Babylonia. The white naphtha,

which attracts fire, is composed of liquid sulfur (?); the black naphtha is simply liquid black asphalt, which can be burnt in lamps instead of olive oil."

He states further (XVI, 743):

"There is much asphalt in Babylonia, about which Erathosthenes (of Alexandria, 276 to 194 B.C.) has the following to say: "The liquid, which is called naphtha, is found in the region around Susa. The more solid material, which may become hard, occurs in Babylonia. The source is near the Euphrates. When this river overflows its banks, through the melting of the snow on the mountains, this well also overflows and wends its way to the river. With it come large lumps of asphalt which is very suitable for use in cementing bricks for house building. Others say that the liquid kind also occurs in Babylonia. I have already said how useful the solid kind is for the building of houses. It is said that ships are also made of it and that if they are coated with it, they will become watertight'."

Strabo refers (XVI, 738) to the use of asphalt mortar, both for constructing walls of houses and for waterproofing tunnels. He observes (VII, 316):

"Asphalt from Rhodes requires more olive oil for cutting, than that from Pieria (Thessalonica)."

He informs us that "pissasphaltos" [derived from the Greek words "pissa" (pix or pitch) and "asphaltos" (asphalt)] [59] occurs in the neighborhood of Epidamnos (or Dirrachion) on the mainland of Albania, stating (VII, 316):

"In the country of the Apolloniates at a place called Nymphaeum, there occurs a rock, at the base of which, fire gushes forth, accompanied by streams of hot water and melted asphalt, and undoubtedly it is the mass of asphalt that burns. This occurrence is found near the summit. The mass carried away by the water is eventually recovered, since it is again converted into asphalt, which as Poseidonius has noted, may be dug out of the earth on a hill in the vicinity."

This most likely alludes to the present-day deposit of Sélénitzia in Albania.

Strabo also refers to the use of asphalt paints, in respect to which he comments (XVI, 739):

"For want of wood for building, only the beams and stays in houses are made of the wood of the date-palm. The stays are stif-

fener with cord made of plaited rushes. Then everything is plastered and painted and the doors are coated with asphalt."

Diodorus Siculus (*about* 50 *A.D.*). This Greek historian [60] also describes the Dead Sea deposit (XIX, t. 98, Chap. 2) as follows:

"It is a large sea which yields up much asphalt and from which a by no means negligible revenue is derived. The sea is about 500 stadia in length and 60 stadia wide. The water stinks and is exceedingly bitter, so that fish cannot live in it, nor do any other creatures occur in it. Although large rivers of very fresh water flow into it, the sea remains bitter. Every year a large quantity of asphalt in pieces more than 3 plethra (110 yards) float in the middle of the sea. The advent of asphalt is heralded 20 days before its arrival, for all around the sea the stench is wafted by the wind over many stadia, and all the silver, gold and copper in the neighborhood become tarnished, but the tarnish disappears again when the asphalt rises to the surface. The district in the vicinity, which is readily inflammable, and which is pervaded by an unpleasant odor, makes the people's bodies ill and they die young." [61]

In connection with the Babylonian asphalt deposits, he states (II, Chap. 12):

"Of all the many wonders occurring in Babylonia the most astounding consists of the large deposits of asphalt which are found there. So much is found that it not only suffices for many and large buildings, but the surplus is used by the populace, who gather large quantities in various localities, which after drying is used as fuel in place of wood. In spite of the large number of inhabitants who thus consume the asphalt, its supply remains as inexhaustible as the water from the wells.

He also alludes to the fact that in constructing the walls of the city of Media, the stones were cemented together with an asphalt mortar, as previously remarked by Herodotus and Xenophon. He refers to the use of asphalt for embalming the dead (XIX, t. 98, Chap. 2), stating:

"The largest portion of the asphalt derived from the Dead Sea is exported to Egypt, where among other uses, it is employed to mummify dead bodies, for without the mixture of this substance with other aromatics, it would be difficult for them to preserve these for a long time from the corruption to which they are liable."

Vitruvius (*about* 50 *A.D.*). The Roman architect Vitruvius, otherwise known as Marcus Vitruvius Pollio [62] refers to the presence of asphalt in the neighborhood of Babylon, which he describes as being of a liquid consistency, and states further (VII, 3 and 8):

"Asphalt is found in Carthage and Ethiopia, and fluid and solid varieties are found in Arabia . . . likewise near Joppa in Syria. . . . In nomad Arabia are lakes of immense size producing much bitumen which is gathered by the neighboring tribes. This is not surprising, because there are many quarries of hard bitumen there. When a spring of water flows through this land, it draws the bitumen with it, and passing along, the water disappears and deposits the bitumen."

He likewise refers (I, 5 and 8) to the Dead Sea occurrence, those at the city of Hit, also to the Albanian deposit, and mentions the use of asphalt mortar for constructing masonry walls at Media.

Pliny the Elder (23 *to* 79 *A.D.*). This Roman naturalist [63] refers to Dead Sea asphalt (XXXV, 178) as "slimy bitumen" and states that a deposit is found in solid form "as an earth" in the vicinity of Sidon, which undoubtedly corresponds to the present Sûk-el-Chan glance pitch mine (loc. cit.). Pliny refers (VI, 99) to another deposit, most likely in the neighborhood of the present town of Bushire in Mesopotamia, stating:

"Here flows the river Granis through Susiana, on the right bank of which the Deximontani dwell, who manipulate bitumen."

Again, he refers (II, 235) to:

"Petroleum wells at Astacensis in Parthia (northeastern Persia) capable of yielding asphalt."

Pliny describes (XXXV, 178) how the natives of Sicily gather asphalt, as follows:

"The inhabitants gather the asphalt from the surface of pools, by stirring with branches, to which it will easily adhere."

He comments (VII, 65) upon the high ductility of good asphalt:

"Asphalt that is elastic and cohesive cannot be split apart, since it adheres to all objects that come in contact with it. It will draw out into long threads when anything is dipped into the sticky mass."

Again (XXXV, 180):

"Bitumen is prized most highly when it is bright and heavy; and becomes less bright when mixed with pitch. It has the same properties as sulfur; it cracks, but heals itself, since the cracks again close up."

He states further that:

"The best quality floats on the surface when the mass is boiled . . . and liquid bitumen is burnt in lamps in place of olive oil."

Pliny alludes to "pissasphaltos" (XXIV, 41; XXXV, 182) as follows:

"A mixture of pitch and asphalt being termed pissasphaltos."

He refers to "ampelitis," i.e. mineral wax (XXXIV, 194), stating:

"Ampelitis greatly resembles bitumen, and like bitumen becomes liquid when it has absorbed oil."

He further informs us that "gagates" (asphaltite or jet) are found in Lycia (XXXVI, 141).

Pliny refers to the production of wood tar (XIV, 122 and 127; XVI, 38 and 52) as follows:

"Stacking a large pile of wood, covering it with a layer of earth or sods and then setting fire to the wood. The tar produced in this manner is drawn off through a drain 16 ells long leading from under the stack."

He also alludes to the production of wood-tar pitch (XVI, 52) as follows:

"The boiling of pitch (Latin: 'picem coquere') derived from wood tar."

Likewise, that (XV, 8; XXIV, 24):

"Tar oil can be obtained by stretching a hide over a cauldron containing boiling pitch, and then wringing out the condensed liquid."

He refers to the use of wood tar and wood-tar pitch for waterproofing pottery (XIV, 134; XV, 62), for calking ships (XVI, 56 and 158), and as a paint for roofs and walls (XXXV, 41; XXXVI, 166).

Pliny refers to the use of asphalt in warfare as follows (CIV):

"The attack of Lucullus on the city of Samosata was repelled with the assistance of burning maltha. . . . In the city of Samosata in Commagene, there is a body of water on the surface of which there was ignited a mass of slime called maltha. When this slime comes in contact with any solid bodies, it adheres to them and follows those that retreat. Thus as the city was besieged by Lucullus, the inhabitants ignited the walls and drove back the soldiers and destroyed their weapons. Experiments have proven that the burning slime can only be extinguished with damp earth."

He comments (XXXIV, 15) on the use of asphalt as a paint:

"The ancients coated their bronze monuments with bitumen, which makes it all the more remarkable to remember that they preferred to cover them with gold. I do not know whether it is a Roman invention, although it is said that it was done at Rome first."

And again (XXXV, 183):

"Copper house utensils are painted with bitumen to make them more resistant to heat and flame. . . . Iron workers use it in their workshops for varnishing iron, for the heads of nails, and for many other purposes."

He also refers (XIV, 20) to the use of asphalt:

"To coat the inside of wine casks and water receptacles."

Pliny describes the use of asphalt for medicinal purposes (XXXV, 180 and 182) and recommends it for curing boils, inflammation of the eyes, coughs, asthma, blindness, epilepsy, etc. He tells us that it was sold under the name mûmîa, which we are informed was actually scraped from the mummies taken from tombs. Its alleged curative properties were explained by the fact that it preserved the dead for so many centuries.[64]

Josephus Flavius (*37 to 95 A.D.*). This Roman historian[65] referred to Dead Sea asphalt stating:

"The changes in the color of the Dead Sea are astonishing, since it alters three times daily and when the sun's rays change their direction they are reflected irregularly."

He also describes the use of bitumen in medicine as a remedy for trachoma, leprosy, gout and eczema.

Plutarch (*about 46 A.D.*). This Greek historian Plutarch[66] informs us:

"A Macedonian named Proxenus, who had charge of the King's equipage, on opening the ground by the river Oxus (present Turkoman Republic) in order to pitch his master's tent, discovered a spring of gross oily liquor, which after the surface was taken off came perfectly clear and neither in taste nor smell differed from olive oil, nor was it inferior to it in smoothness and brightness, though there was no olive tree in that country. It is said indeed, that the water of the Oxus is of so unctuous a quality, that it makes the skin of those who bathe in it smooth and shining."

Tacitus (*55 to 117 A.D.*). The Roman historian states[67] in reference to Dead Sea asphalt:

"Those whose calling is to gather asphalt, draw one end into their boat, whereupon the balance of the mass will follow without effort. This is continued until the vessel is filled, whereupon the viscous mass is cut to pieces. . . . This unusual substance floats in heaps upon the Dead Sea and is either towed or pulled ashore by hand, where it is easily manipulated. When it is sufficiently dried, either by the heat of the sun or by the vapors of the earth, it becomes hard and may be broken up into pieces, as wood or stone, by means of chisels or the force of axes. . . . The hardening may be hastened by wetting with vinegar until it acquires the desired cohesion."

Aelian (*Aelianus Claudius*)—(*about 100 A.D.*). This Roman writer and rhetorician refers to various sources of asphalt.[68]

Dioscorides (*about 150 A.D.*). The Greek physician Dioscorides[69] states (I, 83) that there is a source of asphalt, referred to by him as "Lacus Asphaltites," at Sidon, which is assumed to refer to Siddim, i.e., to the Dead Sea deposit. He states further that:

"Asphalt is found in its liquid state at Acragantium (the present Agrigento) in Sicily. It floats on the surface of the springs. It is a kind of liquid bitumen and is used in lamps in place of olive oil."

He also alludes to Albanian asphalt under the term "pissasphaltos" as follows (I, 84):

"In the vicinity of Epidamnos (i.e. Dirrachion in Albania), there is found a substance called 'pissasphaltos.' It comes down from the Ceraunic mountains and is carried along by the force of the water and deposited on the banks of the rivers, where it is found in lumps. It smells of pitch mixed with asphalt."

In reference to Indian asphalt, presumably referring to the deposit at the Basti river, near Isakhel, India, in the upper Indus Valley, he comments (I, 83):

"Bitumen obtained in India is prized most highly, but they do not disclose the locality where it is found. It is so desirable because of its purple hue, its heavy weight and characteristic strong odor. The dark and impure varieties of asphalt are full of faults, since they have been mixed with pitch, and they come from Phoenicia, Babylon, Zacynthos and Sidon."

He states that asphalt may be refined:

"By boiling off the fluid constituents, which make the material so inflammable."

Dioscorides also informs us that:

"The name mûmîa is given to the drug called 'Bitumen of Judea,' and to the mûmîa of the tombs, found in great numbers in Egypt, and which is nothing more than a mixture which the Byzantine Greeks used formerly for embalming their dead, in order that the bodies might remain in the state in which they were buried, and experience neither decay nor change. Bitumen of Judea is the substance which is obtained from the Asphaltites Lake."

He mentions (V, 181) that "ampelitis" comes from Selenica, and "gagates" from Lycia (V, 146); also that (I, 85):

"Asphalt is recommended as a panacea against skin afflictions."

Dion Cassius (155 *to* 230 *A.D.*). This Roman historian states:[70]

"There is Babylon, Trajan saw the asphalt with which the walls of Babylon had been built. Together with bricks or stones, it produces such strength that the walls made of it are stronger than rock and any kind of iron."

Philostratus (*The Elder*)—(*about* 200 *A.D.*). This Athenian writer[71] refers (I, 23) to asphalt deposits near Ardericca (near the present town of Kirab) in Cissia (Iran). He states that in the land of Cissia:

"The soil is drenched with pitch and is bitter to plant in. . . ."

and warns:

"Against drinking water which has been in contact with bitumen, since this will cause the intestines to close up, owing to the water taking up traces of bitumen."

although he goes on to say ("Tetrabiblos," I, 2 and 49):

"That the drinking of bituminous water is the best remedy for dropsy."

He also describes:

"An oil which once set afire, cannot be extinguished, and which Indian kings use to burn down walls and capture cities."

Geoponica (200 *to* 300 *A.D.*). The "Geoponica" [72] consisted of a collection of writings on husbandry and agriculture by various Greek and Roman writers of the third and fourth centuries, including Gargilius Martialis of Mauretania and Palladius of Rome. In this treatise we are informed that a mixture of bitumen and oil served to alleviate wounds in trees (XIII, 10 and 7); that rings of bituminous mastic were made around tree trunks for protection against ants; that bitumen mixed with sulfur was burned under trees and bushes to kill caterpillars and other harmful insects, also for disinfecting the cages of birds (XIV, 11, 4); that fowls would lay bigger eggs if rubbed with a mixture of bitumen, resin and sulfur (XIV, 11); that a mixture of bitumen and various spices served for the treatment of cattle plague (XVII, 16); and lastly, that bitumen relieved sufferers of diarrhoea (XVII, 16, 1).

Africanus (*about* 300 *A.D.*). This Greek writer, in his treatise "Kestoi" [73] on agriculture, natural history, military science, etc., describes a mixture consisting of:

"Sulfur, natural resin (i.e. asphalt), salt and quicklime, which by careful mixing, the addition of the lime last, and enclosing the whole in a bronze vessel to exclude humidity, air and light, will ignite spontaneously by the simple addition of water or dew."

Ammianus Marcellinus (330 to 395 *A.D.*). This Roman historian states,[74] probably in reference to the region of Quâra and Qúala Shergat in Iraq:

"In Assyria there is asphalt in the lake called Sosingite, in the bed of which the Tigris is absorbed, which flows on underground to rise to the surface again at a great distance away. Naphtha also occurs here; this greatly resembles asphalt and is very viscous and sticky. Even if a very small bird alights on it, it is drawn down, it can no longer fly and it disappears into the depths. Once this kind of substance begins to burn, human intelligence will find no other

means of quenching it than by earth. A big cleft will be seen in these districts from which mortally fatal fumes rise up, the heavy odor of which will kill any living creature coming within its reach."

Theophanes (758 *to* 817 *A.D.*). The Greek writer Theophanes, in his treatise "Chronicles," states:

"The principle of spontaneous ignition by contact with water was brought to its highest practical application by the Greek architect Kallinikos residing in Byzantium about 650 A.D., as a fugitive from the Arabs of the Syrian town of Heliopolis, and who was the real inventor of 'Greek Fire'." [75]

This seems strange, in view of the earlier disclosures by Thucydides (471–401 B.C.), Hannibal (247–183 B.C.), Pliny the Elder (23–79 A.D.), Philostratus (about 200 A.D.), and others.

About 950 A.D. Abu-L-Hasan Masudi. The Persian writer Masudi, in his "Annals" [76] refers to "oleum de gagantis," which is an oily product derived from natural asphalt by a process termed "distillatio per decensorium" which was carried out in two superimposed jars separated by a screen or sieve. The upper jar, filled with the material under treatment, was heated by a fire and the oily distillate allowed to drip through the screen into the bottom jar imbedded in damp soil. Medieval writers frequently refer to rock asphalts and asphaltites under the name "gagates," which was derived from the river Gagas or Gages in Lycia, Asia Minor, at the mouth of which there was stated to be a deposit of hard asphalt. Gagates were used as a means of exorcising evil spirits, in which connection Bishop Marbode of Rennes (1067–1123 A.D.) states in his treatise "Dactyliothecae":

"As an amulet it benefits dropsy, diluted with water it prevents loose teeth from falling out, whilst fumigation with gagates is good for epileptics. It remedies indigestion and constipation, cures magical illusions and evil incantations, and is often used in love potions."

About 985 A.D. Abd Al Mukaddasi. This writer refers [77] to:

"Nafta and earth-pitch from Transoxania, Ferghâna,"
which corresponds to the present Baku region.

About 1248 A.D. Pierre de Joinville. De Joinville, who accompanied King Louix IX on his Sixth Crusade to Damiette, refers [78] to the use of Greek Fire, stating that:

"Every man touched by it believed himself lost, every ship attacked was devoured by flames."

About 1300 A.D. Marco Polo. This Venetian traveler described [79] seepages of liquid asphalt at Baku on the Caspian Sea. He also mentioned the existence of an ancient fire-temple erected about the flaming streams of gas and oil, which we are informed constituted a place of Hindoo pilgrimage.

About 1350 A.D. Sir John Mandeville. This British writer states (Chap. XXX): [80]

"And two myle from Jericho is flom Jordan and you shall wete the Dead Sea is right bitter, and this water casteth out a thing called asphatum, as great pieces as a horse."

1494-1555. Georg Agricola. This well known German metallurgist states: [81]

"Bitumen is produced from mineral waters containing oil, also from liquid bitumen, and from rocks containing bitumen. . . . Liquid bitumen sometimes floats in large quantities on the surface of wells, brooks and rivers, and is collected with buckets or other pots. Small quantities are collected by means of feathers, linen towels and the like. The bitumen easily adheres to these objects, and is collected in big copper or iron vessels and the lighter fractions evaporated by heating. The residual oil is used for different purposes and some people mix it with pitch, others with used axle oil to make it thinner. The bitumen does not harden, even during the time of its heating in the vessels. Rocks which contain bitumen are treated in the same way as those which contain sulfur, by heating them in vessels with a sieve bottom. This, however, is not the common practice, because the bitumen prepared in this way is not very valuable." This process is illustrated in Fig. 23.

Agricola also refers to asphaltic crude-oil seepages at Wietze, near Hannover, Germany, and mentions the use of "heavy petroleum to protect woodwork from rain."

1498. Christopher Columbus. On his third voyage to America on July 31, 1498, Columbus discovered the island of Trinidad, where it has been stated: [82] "He careened his galleons and caulked their storm-racked seams with this natural waterproofing material," referring to asphalt derived from Trinidad Lake.

About 1500. Use of Asphalt in Peru. It has been established that the Incas of Peru constructed an elaborate system of highways, some of which were paved with a composition not unlike modern bituminous macadam.

1535. Discovery of Asphalt in Cuba. G. F. Oviedo y Valdés of Spain describes [83] a spring of semi-liquid asphalt in the Province Puerto Principe, near the coast of Cuba, which was used for painting the hulls of ships. Another occurrence is mentioned on the



From Agricola

FIG. 23.—Smelting Bitumen from Bituminous Rock.

shore of Havana harbor, used for similar purposes. He tells us that asphaltic petroleum was used to protect woodwork and masonry in Cuba as early as 1550 A.D.

1563. Cesar Fredericke. This writer in his publication "Voyage to the East Indies," states:

"These barks of the Tigris have no pumps in them, because of the great abundance of pitch, which they have to pitch them with all. This pitch they have in abundance two days' journey from Babylon. Near unto the river Euphrates, there is a city called Heit (Hit), near unto which city there is a great plaine full of pitch, very mar-

vellous to beholde, and a thing almost incredible that out of a hole in the earth, which continually throweth out pitch into the aire with continuall smoake; this pitch is thrown with such a force that being hot it falleth like as it were sprinckled over all the plaine in such abundance that the plaine is always full of pitch. The Mores and the Arabians of that place say that that hole is the mouth of hell, and in truth it is a thing very notable to be marked. And by this pitch the whole of the people have their benefit to pitch their barks."

1595. Sir Walter Raleigh. Sir Walter Raleigh [84] gives a record of his voyage of exploration to the east coast of South America in 1595, wherein he describes his visit to the Island of Trinidad, and gives an account of the so-called "Pitch Lake," of which he wrote:

"March 22, 1595—At this point called Tierra de Brea or Piche, there is that abundance of stone pitch, that all the ships of the world may be therewith laden from thence, and wee made trial of it in trimming our shippes to be most excellent goode and melteth not with the sunne as the pitch of Norway, and therefore for shippes trading the south parts very profitable."

1599. First Classification of Bituminous Substances. Andreas Libavius refers to the uses of asphalt, and classified it with mineral oil, amber and pitch. He endeavored to trace the connection between asphalt and petroleum, and gives a record of the earliest literature on asphalt including the works of Pliny, Dioscorides, Hippocrates and others.[85]

1608. William Shakespeare. In his play "Pericles" written in 1608, by Shakespeare (1564–1616), occurs the passage: "We have a chest beneath the hatches, caulked and bitumened ready."

1656. Early Dictionary Definition of "Bitumen." In one of the earliest dictionaries of the English language, "Thomas Blount's Glossary," bitumen is defined as:

"A kind of clay or slime naturally clammy, like pitch, growing in certain countries of Asia."

It is interesting to note the connection between this interpretation of the word, and the reference to "slime" and "slimepit" in "Genesis."

1660. John Milton. In "Paradise Lost," written about 1660 by Milton (1608–1674), we read: "Blazing cressets fed with naphtha and asphalts"; and again: "The plain, whereon a black

bituminous gurge boils out from underground—the mouth of hell."

1661. Commercial Production of Wood Tar. The earliest reference to the production of wood tar on a large scale by the dry distillation of wood, occurs in Robert Boyle's "Chemistra Scepticus," 1661. This industry is said to have been first practiced in Norway and Sweden.

1672. First Accurate Description of Persian Asphalt Deposits. Dr. J. Fryer accurately describes the occurrences of asphalt in the East Indies and Persia, in his book "Nine Years' Travels" (1672–1681).[86]

1673. Discovery of Elaterite. The first description of elaterite, originally found at Castleton in Derbyshire, England, under the name "Elastic Bitumen," is given by M. Lister in the *Philosophical Magazine and Journal of Science*, London, 1673 (p. 6179).

1681. Discovery of Coal Tar and Coal-Tar Pitch. In a patent (No. 214) taken out in England on August 19, 1681, by Joachim Becher and Henry Serle, entitled "A new way of makeing pitch, and tarre out of pit coale, never before found out or used by any other," we find the first description of coal tar and coal-tar pitch, as well as their methods of production.

1691. Discovery of Illuminating Gas from Coal. Dr. John Clayton, dean of Kildare, England, experimented with the inflammable gas obtained on heating coal in a closed retort. He filled bladders with this gas and demonstrated that it burnt with a luminous flame.

1694. Discovery of Shale Tar and Shale-Tar Pitch. British Patent No. 330, of 1694 (Jan. 29), entitled "Pitch, tar and oyle, out of a kind of stone from Shropshire," granted Martin Eele, Thomas Hancock and William Portlock, contains the earliest record of the manufacture of shale tar and shale-tar pitch.

1712-1730. Discovery of Val de Travers, Limmer and Seyssel Asphalt Deposits. The asphalt deposit at the Val de Travers in the Jura Mountains, Canton of Neuchâtel, Switzerland, was discovered by the Greek Doctor Eirinis d'Eyrinys in 1712, and described in detail.[87]

Some give Eyrinys the credit of having likewise discovered the Limmer asphalt deposit near Hanover, Germany, in 1730, but this

has not been definitely established. A third discovery of asphalt by Eyrinys, in 1735, at Seyssel in the Rhone Valley, Department of Ain, France, proved to be one of the most important deposits in Europe. This has been worked constantly up to the present time, and will be described later.

1772. First Use of Tar for Flat Roofs. Recent researches ascribe P. J. Marperger of Berngau, near Neumarkt (in the vicinity of Nürnberg, Germany) as the originator of tarred roofs. He described the following method of covering flat roofs: the wooden boards were first caulked with oakum, then sprinkled alternately with tar and a mixture of fine sand with iron slag, until after three or four such treatments a fairly thick protective covering was obtained.[88]

1746. Invention of the Process of Refining Coal Tar. On Dec. 2, 1746, a patent (No. 619) was granted in England to Henry Haskins disclosing: "A new method for extracting a spirit or oil from tar, and from the same process obtaining a very good pitch;" consisting of our present process of fractional distillation in a closed retort connected with a worm condenser.

1752. Samuel Foote. The English dramatist Foote (1720-1777) writes in his book "Taste," published in 1752, of the "salutary application of the asphaltum pot" for preserving the beautifying qualities of the complexion. At this time it was claimed by authorities to be "a sure cure for ringworm, boils, gout, epilepsy, blindness, toothache and colic."

1777. First Exposition of Modern Theory of the Origin of Asphalt. In his "Eléments de Minéralogie," published in 1777 LeSage [89] classified bitumens in the sequence: "Naphtha, petroleum, mineral pitch, maltha and asphalt," and regarded them all as originating from petroleum oil. This closely conforms to the modern views regarding the classification and origin of bitumens.

1788. Discovery of Lignite Tar. Krünitz in 1788 referred to the production of "a tar-like oil" upon destructively distillating "earth coal" (lignite). This was virtually the first description of the manufacture of lignite tar.

1780-1790. Discovery of "Composition" or "Prepared" Roofing. Arvid Faxe of Sweden [90] is given credit for having produced the first composition roofings between the years 1780 and 1790 in

the following crude manner: the roof boards were first covered with plain paper, impregnated with a mixture of copper and iron sulfates, which, after being nailed in place, was coated with heated wood tar to make it waterproof, and then surfaced with various colored mineral earths.

A newspaper published in Leipsic in the year 1791 credits Michael Kag of Mühldorf, Bavaria, with having produced an improved form of prepared roofing by saturating raw paper with varnish, and coating the surfaces with a mineral powder. The product was also recommended as a substitute for leather in the soles of shoes.

Similarly, the *Magdeburg Zeitung* on Nov. 16, 1822, contained a notice stating that paper impregnated with tar is being used to displace straw and wooden shingles for roofing purposes, and that the former may be made fire-resistant by treating the tar with unslaked lime and surfacing with sand.

The manufacture of "tarred board" for roofing purposes, as practiced in Germany during the year 1828, was published by W. A. Lampadius.[91]

1792-1802. Manufacture of Coal Gas and Coal Tar on a Large Scale. Wm. Murdoch, of England, was the first to manufacture coal gas and coal tar on a large scale.

1797-1802. Exploitation of Seyssel Asphalt in France. M. Sécrétan obtained a concession from the French Government to work the asphalt deposits at Seyssel on the Rhone, France, in the fifth year of the French Republic (1797). The venture, however, did not prove a success. The deposit was next taken over by Count de Sassenay, of France, in 1802, and actively exploited. A laboratory was erected to investigate the uses of this asphalt, which was marketed in France under the name "rock asphalt mastic," and used for surfacing floors, bridges and sidewalks, also to a limited extent for waterproofing. The earliest experimental use was in the vicinity of Bordeaux and Lyons.

1815. Commercial Exploitation of Coal-Tar Solvents. In 1815, F. C. Accum, of England, obtained "naphtha" by subjecting coal tar to fractional distillation on a commercial scale. This distillate was used in the manufacture of India rubber goods, for burning in open lamps and for certain kinds of varnish. The tar which

remained behind had no particular value and was accordingly consumed as fuel.

1820. Manufacture of Asphalt-saturated Packing Papers in Switzerland. The asphalt deposits previously discovered at Neuchâtel, Switzerland, were utilized in 1820 for impregnating porous paper, for use as tarpaulins, packing paper, imitation oil-cloth, etc., and the first factory was established in Geneva.[92]

1822. Discovery of Scheererite and Hatchettite. The mineral wax Scheererite was discovered in a bed of lignite (brown coal) at Uznach, near St. Gall in Switzerland, by Captain Scheerer, in 1823.[93] In the same year the mineral wax hatchettite or hatchetine was discovered on the borders of Loch Fyne, in Argyllshire, Scotland, and was named after the English chemist, C. Hatchett.[94]

1830. Discovery of Paraffin Wax. The discovery of paraffin wax is credited to Carl von Reichenbach, of Stuttgart, Germany, who was the first to describe its physical and chemical properties.[95] He derived the material from lignite tar and christened it "paraffin" (parum affinis), because of its unusual resistance to chemicals.

1832. Coal Tar First Used for Paving. The first stretch of tar-macadam pavement on record was laid in Gloucestershire, England, between 1832 and 1838. In this same connection, an interesting pioneer patent was granted to Cassell in 1834, describing the use of coal tar for surfacing roads, grouting, and the construction of tar-concrete paving blocks.[96]

1833. Discovery of Ozokerite. The first reference to the mineral ozokerite was by E. F. Glocker[97] in 1833. He discovered it near the town of Slanik in Moldavia, close to a deposit of lignite at the foot of the Carpathians. It was named from the Greek words signifying "to smell" and "wax," in allusion to its odor.

1835. First Asphalt Mastic Foot Pavements Laid in Paris. It is recorded that on June 15, 1835, the first mastic pavement was laid at Pont Royal, Paris, composed of Seyssel asphalt.[98]

1836. Asphalt First Used in London for Foot Pavements. In 1836 we first hear of Seyssel asphalt being introduced from France to London for constructing foot paths.[99]

1837. Publication of First Exhaustive Treatise on the Chemistry of Asphalt. The well-known treatise "Mémoir sur la composition des bitumes" was published by J. B. Boussingault in the year

1837. It was the most exhaustive treatise on the subject which had yet appeared, and was the first to propose the use of the terms "petrolene" and "asphaltene" for the components of asphalt.[100]

1837. Discovery of Bituminous Matter in the United States. In 1837 appeared the first report of an asphalt deposit in the United States in which semi-solid and solid bitumens were reported in the Connecticut valley at Farmington, Hartford (Rocky Hill), Berlin, Middletown and New Britain, Conn.[101]

1838. Discovery of Process for Preserving Wood with Coal-tar Creosote. In 1838 Bethell disclosed the use of coal-tar oil for impregnating wood.[102]

1838. Asphalt First Used in the United States for Foot Pavements. The earliest case on record of rock asphalt being used in the United States for sidewalks is in the portico of the old Merchants' Exchange Building, Philadelphia, in 1838. Seyssel asphalt was used for this purpose.

1841. First Use of Wood Block Pavement. Wood blocks impregnated with coal tar were first used in England.[103]

1843. Bituminous Matters Discovered in New York State. L. C. Beck, in 1843, wrote a paper on the occurrence of bituminous matter in several of the New York limestones and sandstones.[104]

1844-1847. First Composition Roofing in the United States. Rev. Samuel M. Warren reported that in 1844–1845 roofs were first laid in Newark, N. J., consisting of square sheets of ship's sheathing paper treated with a mixture of pine tar and pine pitch, and surfaced with sand. In 1847 coal tar was used as a substitute for the pine tar, to soften the pine pitch, and employed as a saturant for the paper. Fine gravel was next used to substitute the sand. The square sheets were dipped into the melted mixture by hand, sheet by sheet, and then the excess was pressed out. The next step consisted in running the paper or felt in rolls through continuously operating saturators designed to saturate with tar. Finally, in Buffalo, N. Y., coal tar was distilled down to a roofing pitch, which was used to replace the more expensive mixture of pine pitch and coal tar. The foregoing constituted the origin of the "tar-and-gravel roof," so extensively used to-day.[105]

1850. Discovery of "Asphaltic Coal" in New Brunswick, Nova Scotia. C. T. Jackson published the first accounts of Nova

Scotia "albertite" in the years 1850–1851. It was described as "Albert Coal." [106]

1852. First Modern Asphaltic Road. The first asphaltic road of which we have a record in comparatively modern times, was constructed in 1852 from Paris to Perpignan, France, after the fashion of modern macadam construction, from Val de Travers rock-asphalt.

1854. First Compressed Asphalt Roadway Laid in Paris. In 1854 a short stretch of compressed rock asphalt roadway was laid in Paris by M. Vaudrey.[107] This, we are told, was the outcome of observations previously made by a Swiss engineer, M. Merian, who in 1849 noted that fragments of rock asphalt which fell from the carts transporting the material from the mine at Val de Travers, to the nearby village, became compressed in summer under the wheels into a crude pavement of asphalt. M. Léon Malo in conjunction with M. Vaudrey and M. Homberg thereupon constructed a stretch of roadway compacted with a roller in the Rue Bergère, Paris, which was maintained in good condition for sixty years.[108]

1858. First Modern Asphalt Pavement Laid in Paris. In 1858 the first large area of asphalt roadway was constructed on the Palais Royal and on the Rue St. Honoré in Paris. It was composed of a foundation of concrete 6 in. thick surfaced with rock asphalt mastic obtained from the Val de Travers deposit, compressed to a layer about 2 in. thick. This constituted the earliest use of sheet asphalt pavement in its modern form.

1863. Discovery of Grahamite in West Virginia. The first account of the West Virginia grahamite deposit is given by J. P. Lesley.[109] The material was described as a "rock asphalt," but was later named "grahamite" by Henry Wurtz, in honor of the Messrs. Graham, who were largely interested in the mine.

1869. The First Compressed Asphalt Pavement in London. The first stretch of asphalt roadway in London was laid at Threadneedle Street near Finch Lane in May, 1869. It was composed of Val de Travers rock asphalt.[110]

1870-1873. First Asphalt Roadways in the United States. Some give the Belgian chemist, E. J. De Smedt, credit for having laid the first rock asphalt roadway in the United States, contending

that in 1870 a small experimental strip was laid with continental asphalt opposite the City Hall in Newark, N. J. In 1871 pavements were laid in Washington, D. C., in accordance with a patent granted to N. B. Abbott,[111] composed of a mixture of crushed rock and sand with coal-tar pitch and creosote oil, after a favorable report had been rendered by a special commission appointed by Congress. These pavements gave good service for over 15 years. Also in 1871, a small stretch of pavement was laid in Battery Park, New York City, and according to T. H. Boorman,[112] in 1872 a larger stretch was laid at Union Square, composed of Val de Travers rock asphalt. It is also reported that in 1873 a similar pavement was laid in front of the Worth Monument, which remained in use until 1886.[113]

1876. First Trinidad Asphalt Pavement Laid in the United States. According to Clifford Richardson [114] the first sheet asphalt pavement of Trinidad asphalt to be laid in the United States was on Pennsylvania Avenue, Washington, D. C. This was authorized by an act of Congress passed in 1876, Val de Travers rock asphalt being used from the Capitol to Sixth Street, and Trinidad asphalt for the remainder. The rock asphalt was subsequently pronounced to be too slippery and the Trinidad asphalt pavement a success.

1880. Use of Asphalt "Chewing-gum" in Mexico. B. de Sahagun [115] informs us that the inhabitants of the coastal districts of Mexico, the Totonacs, collected asphalt ("tzacutli") in the Panuco river region and sold it to the Aztecs of the interior, who in turn compounded it with gum chicle ("txixtli") and used the mixture as chewing-gum ("chapopotli").

1881. Use of Chemicals for Oxidizing Coal Tars and Petroleum Asphalts. The first *complete* disclosure of the process for "oxidizing" bituminous materials was by E. J. De Smedt.[116] This process consisted in evaporating coal tar or asphalt in contact with substances capable of inducing oxidation (such as potassium permanganate), "to give them greater tenacity and render them, or the pavement, or other compositions in which they enter, less brittle and less liable to be affected by air or water."

1885. Discovery of Uintaite (Gilsonite) in Utah. Gilsonite, first known as "uintaite," was discovered in the Uinta Valley near Fort Duchesne, Utah, in 1885. It was first described by W. P.

Blake[117] and was later called "gilsonite," after S. H. Gilson, of Salt Lake City.

1889. Discovery of Wurtzilite in Utah. W. P. Blake subsequently discovered a deposit of wurtzilite not far from the source of gilsonite in the Uinta Valley, Wasatch County, Utah, between Salt Lake and the valley of the Green river.[118] It was named after Dr. Henry Wurtz of New York.

1891. Exploitation of the Bermudez Asphalt Deposit, Venezuela. According to Hippolyt Köhler and Edmund Graefe,[119] the Bermudez asphalt deposit in Venezuela was first developed in the year 1891 by the New York-Bermudez Company, and subsequently taken over by the Barber Asphalt Paving Co. A search of the literature fails to reveal when this deposit was first discovered.

1892. Use of Bermudez Asphalt on a Large Scale. Bermudez asphalt was first used extensively in Detroit, Mich., in 1892, and the year following in Washington, D. C. With this start, it rapidly gained in popularity, and is to-day largely used as a road binder, as well as for sheet-asphalt pavements.

1894. Use of Air for Oxidizing Petroleum Asphalt. A further development of the De Smedt process for oxidizing petroleum asphalt was brought about by F. X. Byerley, of Cleveland, O., who blew air through asphaltic oils maintained at a temperature of 600° F.[120] The resulting product, marketed under the name of "byerlite," attained great popularity.

CHAPTER II

TERMINOLOGY AND CLASSIFICATION OF BITUMINOUS SUBSTANCES

One of the most baffling problems with which we have had to deal in recent years is fixing the definitions of the various bituminous substances, and the products in which they are used in the arts.[1]

The words "bitumen," "asphalt," "resin," "tar," "pitch," "wax," have been in use for many centuries, most of them long before the advent of the English language. At first, very little was known regarding the properties of these substances, and, as a result, the early writers used these terms loosely, and, in many cases, interchangeably. It is probable that each of these words at first related to the aggregate characteristics of some typical substance closely associated with the processes of daily life. As nothing of the chemistry was known when these terms originated, they were at first differentiated solely by their *physical* characteristics.

The words originally had but a limited meaning, but as new members of these groups of substances were discovered, the terms were extended in scope until the various expressions completely outgrew their original bounds. This resulted in a certain amount of overlapping and ambiguity.

As the chemistry of these substance gradually became known, this means was likewise adopted to differentiate between them, but we are still compelled to rely principally upon the physical characteristics in arriving at a rational basis of terminology, as their chemistry has been unravelled to but a limited extent.

In defining a substance, we must rely on one or more of the following criteria:

Origin,	Solubility,
Physical Properties,	Chemical Composition.

The last three can be more or less readily ascertained from an examination of the substance itself. The origin, however, is not

always apparent, but in certain cases may be deduced by inference. To base a definition solely upon a statement of the origin of a substance would necessitate some prior knowledge concerning its source or mode of production. As such knowledge is not always available, a definition of this kind would be very limited in its scope. Unfortunately, this plan has often been followed by many of the leading technical societies in this country and abroad, in fixing the definitions of bituminous substances.

A far better method consists in basing the definition upon the *inherent* characteristics of the substance itself, so as to permit of its identification without necessarily having prior knowledge concerning its origin.

The four cardinal features forming this latter basis of nomenclature may be further elaborated as shown in Table I.

In Table II the principal types of bituminous substances are classified according to the features enumerated in Table I.

The definitions which follow are based upon this classification. Although reference is made to the origin of the substance, nevertheless, this is but incidental, and with the exception of the generic terms, the definitions would be explicit even though this feature were omitted.

Bituminous Substances.* A class of native and pyrogenous† substances containing bitumens or pyrobitumens, or resembling them in their physical properties.

SCOPE. This definition includes bitumens, pyrobitumens, pyrogenous distillates and tars, pyrogenous waxes and pyrogenous residues (pitches and pyrogenous asphalts).

Liquid Bituminous Materials. Those having a penetration at 25° C. (77° F.), under a load of 50 g. applied for one second, of more than 350 (Test 9*b*).

Semi-Solid Bituminous Materials. Those having a penetration

* The scope of the word "bituminous" is based on the commonly accepted interpretation of the suffix "ous," signifying: (1) to contain; (2) to resemble, to partake of the nature, to have the qualities (e.g., silici*ous*: containing silica or resembling silica; resin*ous*: containing or resembling resin; oleagin*ous*: containing or resembling oil; calcare*ous*: containing or resembling lime). Similarly, the word "bitumin*ous*" is construed to include substances, either *containing* more or less bitumen (or pyrobitumen), or else *resembling* them in their appearance or qualities.

† The expression "pyrogenous" implies that the substance was produced by means of heat or fire.

TABLE I

Origin	Native	Mineral Vegetable Animal
	Pyrogenous	Factional distillation Destructive distillation Heating in a closed vessel Blowing with air.
Physical Properties	Color in Mass	Light (white, yellow or brown) Dark (black)
	Consistency or Hardness	Liquid Viscous Semi-solid Solid
	Fracture	Conchoidal Hackly
	Lustre	Waxy Resinous Dull
	Feel	Adherent Non-adherent Unctuous (waxy)
	Odor	Oily (petroleum-like) Tarry
	Volatility	Volatile Non-volatile
	Fusibility	Fusible Difficultly fusible Infusible (melts only with decomposition)
Solubility	Non-mineral constituents soluble in carbon disulfide	
Chemical Composition	Hydrocarbons (compounds containing carbon and hydrogen) Oxygenated bodies (compounds containing carbon, hydrogen, and *oxygen*) Crystallizable paraffins (crystallize at low temperatures) Mineral matter (inorganic substances).	

at 25° C. (77° F.), under a load of 100 g. applied for five seconds, of more than 10, and a penetration at 25° C. (77° F.), under a load of 50 g. applied for one second, of not more than 350 (Test 9*b*).

Solid Bituminous Materials. Those having a penetration at 25° C. (77° F.), under a load of 100 g. applied for five seconds, of not more than 10 (Test 9*b*).

TABLE II

Bituminous Substances	Origin	Physical Properties							Solubility	Chemical Composition
		Color in Mass	Consistency	Lustre	Feel	Odor	Volatility	Fusibility, Non-mineral Constituents	Non-mineral Constituents in Carbon Disulfide	
Bitumen.	Native.	Variable (light to dark).	Variable (liquid to solid).	.....	.....	.....	Variable.	Harder varieties are fusible.	Largely soluble.	Yields water-insoluble sulfonation products, substantially free from oxygenated bodies, sometimes associated with mineral matter.
Pyrobitumen.	Native.	Dark.	Comparatively hard.	Resinous to dull.	Non-adherent.	.....	Non-volatile.	Infusible.	Relatively insoluble.	Yields water-insoluble sulfonation products, which may or may not contain oxygenated bodies, sometime associated with mineral matter.
Petroleum.	Native.	Variable. (light to dark).	Liquid.	.....	Oily.	Characteristic ("oily").	Comparatively volatile.	.....	Soluble.	Yields water-insoluble sulfonation products, substantially free from oxygenated bodies.
Mineral wax.	Native or pyrogenous.	Variable (light to dark).	Viscous to solid.	Characteristic "waxy."	Unctuous.	.....	Comparatively non-volatile.	Fusible.	Soluble.	Yields water-insoluble sulfonation products, substantially free from oxygenated bodies, containing considerable crystallizable paraffins, sometimes associated with mineral matter.
Asphalt.	Native or pyrogenous.	Dark.	Variable.	Harder varieties resinous to dull.	Adherent to non-adherent.	.....	Comparatively non-volatile.	Fusible.	Largely soluble.	Yields water-insoluble sulfonation products, substantially free from oxygenated bodies, containing little to no crystallizable paraffins, sometimes associated with mineral matter.
Asphaltite.	Native.	Dark.	Comparatively hard.	Resinous.	Non-adherent.	.....	Non-volatie.	Difficultly fusible.	Largely soluble.	Yields water-insoluble sulfonation products, substantially free from oxygenated bodies and crystallizable paraffins, sometimes associated with mineral matter.
Asphaltic pyrobitumen.	Native.	Dark.	Comparatively hard.	Resinous to dull.	Non-adherent.	.....	Non-volatile.	Infusible.	Largely insoluble.	Hydrocarbons, substantially free from oxygenated bodies, sometimes associated with mineral matter.
Non-asphaltic pyrobitumen.	Native.	Dark.	Comparatively hard.	Resinous. to dull.	Non-adherent.	.....	Non-volatile.	Infusible.	Largely insoluble.	Hydrocarbons, containing oxygenated bodies, sometimes associated with mineral matter.
Tar.	Pyrogenous.	Dark.	Liquid.	.....	Oily.	Characteristic ("tarry").	Comparatively volatile.	.....	Largely soluble.	Yields water-soluble sulfonation products. Variable composition.
Pitch.	Pyrogenous.	Dark.	Viscous to solid.	Resinous to dull.	Adherent to non-adherent.	.....	Comparatively non-volatile.	Fusible.	Largely soluble.	Yields water-soluble sulfonation products. Variable composition.

Bitumen.* A generic term applied to native substances of variable color, hardness and volatility; composed principally of hydrocarbons, substantially free from oxygenated bodies; sometimes associated with mineral matter, the non-mineral constituents being *fusible* and largely *soluble* in carbon disulfide, yielding water-insoluble sulfonation products.

SCOPE. This definition includes petroleums, native asphalts, native mineral waxes and asphaltites (gilsonite, glance pitch and grahamite).

Pyrobitumen.† A generic term, applied to native substances of dark color; comparatively hard and non-volatile; composed of hydrocarbons, which may or may not contain oxygenated bodies; sometimes associated with mineral matter, the non-mineral constituents being *fusible* and relatively *insoluble* in carbon disulfide.

SCOPE. This definition includes the asphaltic pyrobitumens (elaterite, wurtzilite, albertite and impsonite) also the non-asphaltic pyrobitumens (peat, lignite, bituminous coal and anthracite coal) and their respective shales.

Petroleum. A species of bitumen, of variable color, *liquid* consistency, having a characteristic odor; comparatively volatile; composed principally of hydrocarbons, substantially free from oxygenated bodies; soluble in carbon disulfide, yielding water-insoluble sulfonation products.

SCOPE. This definition includes non-asphaltic, semi-asphaltic and asphaltic petroleums.

Mineral Wax. A term applied to a species of bitumen, also to certain pyrogenous substances; of variable color, viscous to solid consistency; having a *characteristic lustre* and *unctuous feel;* comparatively non-volatile; composed principally of saturated hydrocarbons, substantially free from oxygenated bodies; containing *considerable crystallizable* paraffins; sometimes associated with mineral

* The interpretation of the term "bitumen" as employed in this treatise is entirely dissociated from the idea of *solubility* (in certain solvents for hydrocarbons), and has no connection whatsoever with the inappropriate expression "total bitumen," used in many contemporary text-books to designate the amount soluble in carbon disulfide, and which unfortunately is largely responsible for the existing confusion in terminology.

† The expression "pyrobitumen" implies that the substance when subjected to heat or fire will generate, or become transformed into bodies resembling bitumens (in their solubility and physical properties).

matter, the non-mineral constituents being easily fusible and soluble in carbon disulfide, yielding water-insoluble sulfonation products.

SCOPE. This definition is applied to crude and refined native mineral waxes, also to pyrogenous waxes. Crude native mineral waxes include ozokerite. Refined native mineral waxes include ceresine (refined ozokerite) and montan wax (extracted from lignite or pyropissite by means of solvents). Pyrogenous waxes include the solid paraffins separated from non-asphaltic and semi-asphaltic petroleums, peat tar, lignite tar and shale tar.

Asphalt. A term applied to a species of bitumen, also to certain pyrogenous substances of *dark color*, variable hardness, comparatively non-volatile; composed principally of hydrocarbons, substantially free from oxygenated bodies; containing relatively little to no crystallizable paraffins; sometimes associated with mineral matter, the non-mineral constituents being *fusible*, and largely soluble in carbon disulfide, yielding water-insoluble sulfonation products.

SCOPE. This definition is applied to native asphalts and pyrogenous asphalts. Native asphalts include asphalts occurring naturally in a pure or fairly pure state, also asphalts associated naturally with a substantial proportion of mineral matter.* The associated mineral matter may be sand, sandstone, limestone, clay, shale, etc. Pyrogenous asphalts include residues obtained from the distillation, blowing, etc., of petroleums (e.g., residual oil,† blown asphalts,‡ residual asphalts,§ sludge asphalt,|| etc.), also from the pyrogenous treatment of wurtzilite (e.g., wurtzilite asphalt ¶).

In Europe, the term "asphalte" is applied to unconsolidated limestone impregnated with asphalt, which softens and crumbles when subjected to a moderate heat (e.g., 125° to 160° F.), whereas the term "bituminous rock" is used to designate consolidated limestone rock impregnated with asphalt, which resists high temperatures (e.g., 1000° F. and over) without crumbling.

Asphaltite. A species of bitumen, including dark-colored, comparatively hard and non-volatile solids; composed principally of

* Often termed "rock asphalts."

† Produced by the dry distillation of non-asphaltic petroleum, the dry or steam distillation of semi-asphaltic petroleum and the steam distillation of asphaltic petroleum.

‡ Produced by blowing air through heated residual oils.

§ Produced by the steam distillation of semi-asphaltic and asphaltic petroleums.

|| Produced from the acid sludge obtained in the purification of petroleum distillates with sulfuric acid.

¶ Produced by depolymerizing wurtzilite in closed retorts.

hydrocarbons, substantially free from oxygenated bodies and crystallizable paraffins; sometimes associated with mineral matter, the non-mineral constituents being *difficultly fusible*, and largely soluble in carbon disulfide, yielding water-insoluble sulfonation products.

SCOPE. This definition includes gilsonite, glance pitch, and grahamite.

Asphaltic Pyrobitumen. A species of pyrobitumen, including dark-colored, comparatively hard and non-volatile solids; composed of hydrocarbons, *substantially free from oxygenated bodies*; sometimes associated with mineral matter, the non-mineral constituents being *infusible* and largely *insoluble* in carbon disulfide.

SCOPE. This definition includes elaterite, wurtzilite, albertite, impsonite and the asphaltic pyrobituminous shales.

Non-asphaltic Pyrobitumen. A species of pyrobitumen, including dark-colored, comparatively hard and non-volatile solids; composed of hydrocarbons, *containing oxygenated bodies*; sometimes associated with mineral matter, the non-mineral constituents being *infusible*, and largely *insoluble* in carbon disulfide.

SCOPE. This definition includes peat, lignite, cannel coal, bituminous coal, anthracite coal, and the non-asphaltic pyrobituminous shales.

Tar. A term applied to pyrogenous condensates obtained in the destructive distillation of organic materials; of dark color, *liquid* consistency; having characteristic odors; comparatively volatile at high temperatures; composed principally of hydrocarbons, sometimes associated with carbonaceous matter, the non-carbonaceous constituents being largely soluble in carbon disulfide, yielding water-soluble sulfonation products.

SCOPE. This definition includes the volatile oily decomposition products obtained from the pyrogenous treatment of petroleum (water-gas tar and oil-gas tar), bones (bone-tar), wood and roots of coniferae (pine tar), hardwoods, such as oak, maple, birch, and beech (hardwood tar), peat (peat tar), lignite (lignite tar), bituminous coal (gas-works coal tar, coke-oven coal tar, blast-furnace coal tar, producer-gas coal tar, etc.), and pyrobituminous shales (shale tar).

Pitch. A term applied to pyrogenous residues obtained in the distillation of organic materials; *of dark color*, viscous to solid consistency; comparatively non-volatile, fusible; composed principally of hydrocarbons; sometimes associated with carbonaceous matter, the non-carbonaceous constituents being largely soluble in carbon disulfide, yielding water-soluble sulfonation products.

SCOPE. This definition includes residues obtained from the distillation of tars (oil-gas-tar pitch, water-gas-tar pitch, bone-tar pitch, wood-tar pitch, peat-tar pitch, lignite-tar pitch, gas-works coal-tar pitch, coke-oven coal-tar pitch, blast-furnace coal-tar pitch, producer-gas coal-tar pitch, and shale-tar pitch); also from the distillation of fusible organic substances, the process having been terminated before the formation of coke (rosin pitch and fatty-acid pitch); also anthracene pitch, naphthol pitch, cresol pitch, ozokerite pitch, montan pitch, rubber pitch, gutta-percha pitch, etc., known in Germany as "Immediate Pitches," i.e., produced directly (immediately) on distillation, without the initial formation of tars.[2]

In Germany, the terms "Chemopeche" and "Chemoasphalte" are used to designate pitches and asphalts, respectively, which are produced as the result of chemical reactions, including the following:[3]

(a) Precipitation products, resulting from the precipitation or selective extraction by means of solvents.

(b) Oxygenated products, resulting from the treatment with air, oxygen, ozone, etc., at elevated temperatures, e.g. blown petroleum asphalt, blown coal-tar pitch, etc.

(c) Hydrogenated products, resulting from treatment with hydrogen in the presence of a catalyst at elevated temperatures, and distillation of the resultant product.

(d) Reaction with mineral acids (i.e., sulfuric, nitric, phosphoric, etc.) and evaporation or distillation of the residue, e.g. sludge asphalt.

(e) Reaction with alkalies and subsequent precipitation, extraction, or distillation of the resultant product.

(f) Reaction with sulfur or sulfur derivatives at elevated temperatures, e.g. sulfurized asphalts and pitches.

(g) Reaction with halogens (e.g. chlorine, iodine, etc.).

(h) Reaction with phosphorus and its derivatives.

(i) Reaction with metallic salts; either solid salts at elevated temperatures, or aqueous solutions of metallic salts in refining operations, and separation of the resultant polymerized product.

(j) Reaction with sundry chemicals (e.g. formaldehyde and its derivatives, furfurol, etc.).

It will be noted that the terms "asphalt" and "mineral wax" are each applied indiscriminately to native and pyrogenous substances. This is due to the fact that at the present time it is practically impossible to distinguish between certain native and pyrogenous asphalts or mineral waxes, either by physical or chemical means. It is probable that some method may be discovered for accomplishing this, in which event it would be of decided advantage to frame separate definitions to distinguish between native and pyrogenous substances respectively. With the knowledge available at present, however, this cannot readily be accomplished. We must be content, therefore, to apply the terms "asphalt" and "mineral wax" both to native substances and to manufactured (pyrogenous) products.

In many of the early classifications, natural gas and marsh gas were included within the scope of the term "bitumen." As this stretches the meaning to an abnormal extent, the author deems it inadvisable to include natural gases in the definitions and classifications given in this treatise.

The terms "maltha" (derived from the Greek μάλθα), "brea" and "chapapote" (of Mexican Spanish origin), "goudron minérale" (French), "Bergteer" (German), "kir" (Russian),[4] and "mineral tar" (English), frequently found in contemporary classifications to designate the softer varieties of native asphalt, have been omitted for the sake of brevity.

When it comes to classifying bituminous substances, the interpretation of the word "bitumen" represents the crux of the entire matter. An analysis of the views concerning the scope of this term, prevalent in America and abroad, shows that they may be grouped into four classes, which will be listed in the order of their breadth, commencing with the one having the narrowest scope:

(*a*) *Bitumens*—naturally occurring hydrocarbons.
(*b*) *Bitumens*—naturally occurring hydrocarbons, likewise residues obtained from the distillation of petroleum (e.g., petroleum asphalts).
(*c*) *Bitumens*—naturally occurring hydrocarbons, likewise residues obtained from the distillation of petroleum, likewise *artificial* hydrocarbon substances (e.g., tars and pitches).

(*d*) *Bitumens*—only those components of (*c*) which are soluble in carbon disulfide.

View (*a*) represents the layman's interpretation of the word, as is reflected in the principal dictionaries of both abroad and America. It is obvious from these definitions that the word "bitumen" has for generations been confined to hydrocarbon substances which occur in *nature,* and distinctly excludes hydrocarbon substances produced artificially.

View (*b*) is supported by the British Engineering Standards Association, also by Dr. Heinrich Mallison in Germany. This involves a slight departure from View (*a*), including as it does, residues obtained from the distillation of petroleum.

View (*c*) is still somewhat broader than (*b*), being extended to include tars and pitches. It is supported abroad by David Holde and Wilhelm Reiner and others.

View (*d*) has been adopted in principle by the American Society for Testing Materials,[5] and the American Standards Association.

Now the question arises as to whether there is any need or justification of extending the dictionary definitions embodied in View (*a*). In other words, has the scientific fraternity the right to *change* or modify what has been the common heritage of the people, without some particularly good reasons? The only apparent justification for extending the scope of the word "bitumen" as reflected in Views (*b*) and (*c*) is to include certain products which have been developed and produced since the word was originally adopted—such products as petroleum asphalts, tars and pitches. Parenthetically, there seems to be but little justification to extend the word "bitumen" to include petroleum asphalts (as outlined in View *b*), since the process of distillation has a marked effect on the nature of the asphalt originally present in the petroleum. At the high end-temperatures at which the distillation process is conducted (600° to 800° F.) a form of polymerization takes place, whereby asphalt-like substances are produced. In other words, the percentage of asphalt in the petroleum is *increased* under these conditions. It follows therefore, that the petroleum asphalts produced commercially, differ in their physical and chemical properties from the asphalt originally present in the petroleum, or separated in nature by a slow natural process of *evaporation.*

If the word "bitumen" is broadened in accordance with View (*c*)

it will become synonymous with the word "bituminous substance," so that both expressions will have an identical meaning.

Singularly, there seems to be but very little difference of opinion at the present time with respect to the expression "bituminous substance." In this, the scientific fraternity seems to be in accord with the dictionary definitions. It is the consensus of opinion that the expression "bituminous substance" represents a form of substance which *contains* bitumens, or *resembles* bitumens, or *constitutes the source* of bitumens. This is based on a commonly accepted interpretation of the suffix "ous," signifying:

1—to contain;
2—to resemble, to partake of the nature, or to have the qualities of.

This raises the question as to whether or not language as it exists at present does not furnish us with sufficient tools to answer our needs. Is not the expression "bituminous" ("bituminous substance") of sufficient breadth to include "bitumens" (i.e., naturally occurring hydrocarbons) *as well as* petroleum asphalts, tars and pitches? Is it not true that petroleum asphalts, tars and pitches *resemble* "bitumens" or *partake of their nature* in respect to their physical properties? Then why broaden the scope of the word "bitumen" so as to make it synonymous with the expression "bituminous substance"? The author can see no justification or necessity for so doing.

Moreover, View (*d*) is so technical, that it is questionable whether the laity will ever accept it. Its proponents maintain that the only way to determine whether or not a substance is a "bitumen" is to subject it to chemical analysis, to find out what proportion is soluble in carbon disulfide. According to this view, the presence of any associated mineral constituents or hydrocarbons insoluble in carbon disulfide would disqualify the *original* substance as a "bitumen"—even though it occurred as such *in nature.*

The author therefore advocates that the words "bitumen" and "bituminous substance" be defined in scope as expressed in View (*a*), which is exactly as they have been heretofore used and accepted in the language. He believes further that by using these two terms co-jointly, a satisfactory system of classification may be devised, which will answer adequately the needs of the scientific fraternity,

TABLE III

Classification of Bituminous Substances

Genus	Species	Member	Remarks
Bitumens	Petroleums	Non-asphaltic petroleum..	Contains an appreciable quantity of crystallizable paraffin and no asphalt.
		Semi-asphaltic petroleum.	Contains crystallizable paraffin, also asphalt.
		Asphaltic petroleum.....	Contains an appreciable quantity of asphalt and no crystallizable paraffin.
	Native mineral waxes	Ozokerite	A paraffinaceous mineral, called ceresine when refined.
		Montan wax	The wax extracted from lignite or pyropissite by means of solvents.
	Native asphalts	Pure or fairly pure.....	Comparatively free from associated mineral matter (less than 10 per cent on the dry weight.)
		Associated with mineral matter	Containing a substantial proportion of sand, sandstone, limestone, clay or shale, or filling the voids in a matrix of rock (i.e., "rock asphalt.")
	Asphaltites	Gilsonite.....	Extremely pure } Have a higher fusing-point than asphalts—derived from petroleum.
		Glance pitch.....	Pure to moderately pure }
		Grahamite.....	Pure to quite impure }
Pyrobitumens	Asphaltic pyrobitumens	Elaterite.....	Rubbery—partly saponifiable } Generally pure. Infusible and insoluble. Derived from petroleum.
		Wurtzilite.....	Depolymerizes on heating, becoming fusible and soluble }
		Albertite.....	Depolymerizes partially on heating }
		Impsonite.....	Does not depolymerize on heating. }
		Asphaltic pyrobituminous shales.....	Mineral matters predominate. Infusible and insoluble.
	Non-asphaltic pyrobitumens	Peat.....	Pure or fairly pure. Infusible and insoluble. Contain more or less oxygenated bodies. Derived from vegetable growths. Gradual transition from peat to lignite to coal.
		Lignite.....	
		Bituminous coal.....	
		Anthracite coal.....	
		Lignitic and coal shales...	Mineral matters predominate, otherwise the same as the foregoing.

Pyrogeneous distillates	Pyrogenous waxes	Wax tailings	Distillate from petroleum obtained immediately prior to coking.
		Petroleum paraffin	Solid paraffin obtained from non-asphaltic petroleum.
		Peat paraffin	Solid paraffin obtained from peat tar.
		Lignite paraffin	Solid paraffin obtained from lignite tar.
		Shale paraffin	Solid paraffin obtained from shale tar.
	Tars	Oil-gas tar	Produced by cracking petroleum vapors in manufacturing oil gas.
		Water-gas tar	Produced by cracking petroleum vapors in manufacturing carburetted water gas.
		Pine tar	Produced by the destructive distillation of the wood and roots of coniferae.
		Hardwood tar	Produced by the destructive distillation of hardwoods.
		Peat tar	Produced by the destructive distillation of peat.
		Lignite (brown coal) tar	Produced by the destructive distillation of lignite (brown coal).
		Shale tar	Produced by the destructive distillation of pyrobituminous shales.
		Gas-works coal-tar	Produced from gas-house retorts in manufacturing gas from bituminous coal.
		Coke-oven coal-tar	Produced from by-product coke-ovens in manufacturing coke from bituminous coal
		Blast-furnace coal-tar	Produced from blast-furnaces upon smelting metals with bituminous coal.
		Producer-gas coal-tar	Produced from gas-producers in manufacturing producer gas from coal.
		Bone tar	Produced by the destructive distillation of bones.
Pyrogenous residues	Pyrogenous asphalts	Residual oils	Produced by the dry distillation of non-asphaltic petroleum, the dry or steam distillation of semi-asphaltic petroleum or the steam distillation of asphaltic petroleum.
		Pressure tars	Produced from petroleum by cracking at high temperatures under pressure.
		Blown petroleum asphalts	Produced by blowing air through heated residual oils.
		Residual asphalts	Produced by the steam distillation of semi-asphaltic and asphaltic petroleums.
		Sludge asphalts	Produced from the acid sludge obtained in the purification of petroleum distillates with sulfuric acid.
		Wurtzilite asphalt	Produced by depolymerizing wurtzilite in closed retorts.
	Pitches	Oil-gas-tar pitch	Residues obtained by the partial evaporation or distillation of the corresponding tars.
		Water-gas-tar pitch	
		Wood-tar pitch	
		Peat-tar pitch	
		Lignite-tar pitch	
		Shale-tar pitch	
		Gas-works coal-tar pitch	
		Coke-oven coal-tar pitch	
		Blast-furnace coal-tar pitch	
		Producer-gas coal-tar pitch	
		Bone-tar pitch	
		Rosin pitch	Residue obtained by the partial distillation of the resinous sap of coniferae.
		Fatty-acid pitch	Residue obtained by the steam distillation of fatty-acids.

and at the same time be in complete harmony with the dictionary definitions as they now exist.

With the foregoing in mind, the author has worked out a basis for classifying bituminous substances, including the most important members recognized commercially, which will be found in Table III.

CHAPTER III

CHEMISTRY OF BITUMINOUS SUBSTANCES

Bituminous substances in general may be regarded as consisting of one or more of the following components:

(A) The Non-mineral Matrix.
(B) Associated Mineral Constituents.
(C) Associated Non-mineral Constituents.

Each component will be considered in turn.

(A) COMPOSITION OF NON-MINERAL MATRIX

The non-mineral matrix present in bituminous substances is a complex mixture of hydrocarbons together with their sulfur and nitrogen derivatives, and are frequently associated with mineral constituents in varying amounts. The non-mineral constituents are accordingly composed of the elements carbon and hydrogen, with more or less sulfur, nitrogen, and at times oxygen. The constituent hydrocarbons may either be saturated or unsaturated. Saturated hydrocarbons are those in which the carbon valence has been completely taken up by hydrogen or other radicals, and are characterized by their resistance to reagents (e.g. acids and alkalies) and the difficulty with which they form substitution compounds. Unsaturated hydrocarbons are those having free carbon valences, and have the property of forming additive compounds.

Every member of the bituminous family is a homogeneous or heterogeneous mixture, consisting of a multitude of chemical substances, each having a definite molecular composition. These constituent substances may be associated as a simple solution of liquids in liquids, or solids in liquids; or in the form of a colloidal solution; or as a solid solution of amorphous or crystalline solids; or as an emulsion of immiscible liquids; or as a suspension of insoluble substances in a more or less liquid matrix; or combinations of two or more of the foregoing phases.

It is contended that the characteristic dark color of asphalts is due to the liberation of colloidal carbon under the influence of heat, which is then adsorbed by the hydrocarbons.[1] The colloidal nature of asphalt is confirmed by the Tyndall effect, the Brownian movement observed under the ultra-microscope even in dilute solutions, the fact that on distillation no trace of asphaltic or coal-like substances are found in the distillate, and the further fact that asphaltenes retain hydrogen even at 800° F.

F. J. Nellensteyn regards asphalt as a protected "lyophobe sol," or an extremely stable "carbon-oleosole," in the form of a "system" consisting of three components, viz.:

(1) The "medium" (corresponding to the so-called "petrolenes" or "oily constituents").

(2) The "lyophilic portion" or protective bodies (corresponding to the so-called "asphaltic resins").

(3) The "lyophobic portion," composed of colloidal particles or ultra-microns of elemental carbon.

An adsorption relation exists between components (2) and (3), forming a "disperse phase" and constituting the so-called "asphalt micelle" (corresponding to the "asphaltenes"). According to this hypothesis, the asphalt constituents may be classified into the following systems:

Petrolenes (malthenes) and oily constituents = oily medium.
Asphaltous acids plus asphaltous acid anhydrides plus asphaltic resins = small amount of carbon with very large amount of protective bodies.
Asphaltenes = carbon with protective bodies.
Carbenes = carbon with small amount of protective bodies.
Pyrobitumens = carbon with very little protective bodies.

The stability of the "system" depends upon the respective surface tensions of the "medium" and "micelle." Changes in the stability, including "flocculative" and "peptizing" reactions, give rise to a "reversible flocculation." If, however, the micelle itself is destroyed and cannot be directly repeptized, the substance is said to have undergone an "irreversible flocculation." The latter is caused by chemical reactions (e.g. iodine, chlorine, etc.); by heat; or by exhaustive extraction of asphaltenes with different solvents of successively higher surface-tension. In natural asphalts associated with

colloidal mineral matter (e.g. Trinidad asphalt), the ultra-microns present in the micelle consist partly of elemental carbon and partly of inorganic material. Adding finely divided fillers to asphalt may result in the formation of such micelles.

The properties of asphalts depend upon the concentration of the disperse phase, its degree of subdivision, as well as the properties of the medium. The combination of these functions leads from the crude petroleum, through the soft and viscous asphalts, over to the asphaltites. For the mechanism of this colloidal system in its transition from petroleum to the hard asphalts and asphaltites, the following theory may be developed, in which the underlying idea is clearly illustrated. In Fig. 24 the black points and areas represent the "asphalt micelles," the white points and areas the "oily medium." In region A, the asphalt micelles are present in such slight amount and in such a high degree of subdivision that they exist in the oily medium in the form of a molecularly dispersed solution. This region corresponds to the characteristics of crude petroleum before it is subjected to distillation in the preparation of residual asphalt. In the distillation process, the concentration of the oily medium gradually diminishes, and the particle size of the dispersed phase (asphalt micelles) correspondingly increases, until they assume a dimension larger than molecular, and eventually reach the state of a colloidal dispersion. The gradual enlargement of the micelles, or the increase in their number (through continued distillation) is illustrated by regions B, C, D and E, which represent the disperse systems of soft to moderately hard asphalts. Further reduction of the oily phase causes constantly greater reduction of the distances of the micelles from one another, and accordingly leads to the agglomeration of the asphalt micelles, as shown in region F. We then come into the range of hard asphalts and the reversal of

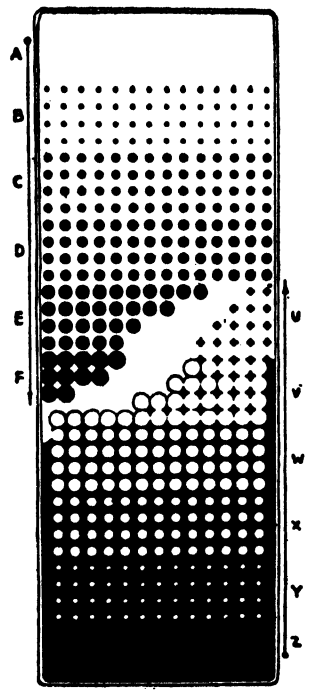


FIG. 24.—Formation of Asphalts Illustrated Diagrammatically.

phases. If the reduction of the oily medium continues further, then the aggregates of the micelles become so large and their number increases to a degree that they eventually fuse together, resulting in the asphalt micelles forming the continuous phase and the oily constituents the disperse phase, as illustrated in regions V, W, X and Y. If the process continues sufficiently far, the system will finally consist only of asphalt micelles, as shown in region Z. Regions V to Z embrace the hard, high fusing-point asphalts and asphaltites.

It is a comparatively simple matter to ascertain by analytical methods the percentage by weight of the elements present. This is termed the *ultimate analysis*, in contra-distinction to the *molecular composition.*

The greatest percentage of carbon found in any bituminous substance is in the case of anthracite coal, which runs as high as 98 per cent. The smallest percentage is contained in certain peats, which run as low as 50 per cent. With the exception of the non-asphaltic pyrobitumens, carbon ranges from 85 to 95 per cent.

Hydrogen never exceeds 15 per cent. In the paraffin series of hydrocarbons, the carbon is combined with as much hydrogen as possible, and this accordingly contains the largest percentage of hydrogen and the smallest percentage of carbon. The lowest member of this series CH_4, a gas, contains 75 per cent of carbon and 25 per cent of hydrogen. The member $C_{30}H_{62}$ contains 85.31 per cent of carbon and 14.69 per cent of hydrogen. In the olefin series C_nH_{2n}, the relation of carbon to hydrogen is constant, and figures: carbon 85.71 per cent and hydrogen 14.29 per cent.

The percentage of sulfur varies considerably. Waxes, coal tar and coal-tar pitch, pine tar and pine-tar pitch, hardwood tar and hardwood-tar pitch, also fatty-acid pitch are practically free from sulfur. In petroleum, the sulfur varies from a trace to 5 per cent as a maximum. Mexican petroleum contains between 3 and 5 per cent; Trinidad, Venezuela and California petroleums contain between ½ and 4 per cent; semi-asphaltic petroleums including the Mid-continental and Texas oils contain from a trace to 2½ per cent. Paraffinaceous petroleums contain merely a trace. Residual oils contain from a trace to 5 per cent. Residual asphalts, blown asphalts, sludge asphalts, native asphalts, asphaltites, asphaltic pyrobitumens and non-asphaltic pyrobitumens contain from a trace to 10

per cent sulfur. Tars and pitches derived from non-asphaltic pyrobitumens contain from a trace to 1½ per cent.

Nitrogen is rarely present in excess of 2 per cent. Mineral waxes are free from nitrogen. Petroleum asphalt, asphaltites and pyrobitumens contain from a trace to 1.7 per cent nitrogen. Tars and pitches contain from 0 to 1 per cent.

Oxygen rarely exceeds 5 per cent, except in the case of non-asphaltic pyrobitumens which contain up to 45 per cent of oxygen.

At the present time but a comparatively small number of distinct chemical substances have been identified in bituminous complexes. A vast amount of research work must yet be accomplished. Although hundreds of substances of definite molecular composition have been identified in petroleums, native mineral waxes, pyrogenous waxes and certain tars, comparatively little is known regarding the innumerable non-mineral molecular substances present in native asphalts, asphaltites, asphaltic pyrobitumens, non-asphaltic pyrobitumens, pyrogenous asphalts and pitches.

The chemistry of bituminous substances is further complicated by the fact that commercial specimens of any given material are rarely alike in composition. In some, certain chemical bodies predominate; in others, they may be present in smaller amounts; while in still others they may be absent. Thus two shipments of any given member of the bituminous family are apt to fluctuate widely in composition and physical properties, even when emanating from the same source. Again, a native bituminous substance derived from a single deposit will often vary, depending upon the degree of exposure and extent of metamorphosis. Native bituminous substances are in a constant state of transition, as the result of their age and environment. Pyrogenous bituminous substances show a marked variation in composition and physical properties, depending upon the raw materials used in their production and the exact conditions to which they have been subjected in their processes of manufacture, including the temperature, length of treatment, etc. Bituminous substances should not therefore be compared with vegetable or animal fats or oils, which in the case of any given material will run fairly uniform in composition and physical properties.

In certain instances, comparatively simple tests have been devised for identifying single chemical bodies present, whereas in other

cases the ultimate analysis of the material will furnish a clue to the identity of the substance under examination.

Various methods have been proposed to separate bituminous substances into their constituents, including fractional distillation under reduced pressure,[2] the selective action of solvents,[3] the use of adsorption media,[4] and combinations of the foregoing.

(B) COMPOSITION OF ASSOCIATED MINERAL CONSTITUENTS

The mineral constituents may be present in one or more of the following typical forms:

1. As consolidated mineral particles consisting of a porous rock impregnated with the bituminous constituents. This type is exemplified by the so-called "rock asphalts," which are usually composed of a fine-grained limestone or sandstone matrix, carrying the asphalt in its voids.

2. As unconsolidated mineral particles admixed mechanically with the bituminous constituents. This is typified by the numerous deposits of impure native asphalts and asphaltites, in which the bituminous constituents are associated with more or less detritus derived from the surrounding soil; also blast-furnace tar and pitch which carry a proportion of mineral dust carried over mechanically by the furnace gases as well as metallic compounds which result from the corrosion of pipe lines and containers used in storage and transport.

The principal unconsolidated mineral constituents present in native asphalts and asphaltites consist of calcium carbonate, magnesium carbonate, calcium sulfate, dolomite, clay, silica and the various silicates, iron sulfide, etc.

3. As colloidal mineral particles held in suspension by the bituminous constituents. Trinidad Lake asphalt is typical of this group, and is characterized by the presence of colloidal clay and silica which are not removable by filtration and are readily discernible when viewed under an ultra-microscope.[5] Richardson makes a special virtue of the fact that refined Trinidad asphalt contains naturally about 27 per cent filler composed largely of "colloidal" particles.

4. As mineral matter held in chemical combination by the non-mineral (i.e., pure bituminous) constituents. This group differs from the foregoing, inasmuch as it relates to a *chemical* union of the mineral and non-mineral components. Many native asphalts carry small percentages of iron and aluminium, but it is as yet a mooted question whether these are present as colloidal particles, or united

chemically with the bituminous matter. Most residual and blown petroleum asphalts contain a trace of iron, derived from the stills in which they are refined. Fatty-acid pitch, wood-tar pitch, bone-tar pitch and rosin pitch carry a substantial amount of iron or copper, depending upon whether they have been produced in an iron or copper still. Sludge asphalts bear a trace of combined lead derived from the lead containers in which they have been treated.

Rare elements are also normally associated with certain native asphalts and asphaltites. Thus the following proportions of nickel, expressed in parts per million, have been reported:[6] Trinidad asphalt 194, Swiss asphalt 120, gilsonite 133, residual asphalt from U. S. petroleum 240, crude U. S. petroleums 1 to 93 parts, ozokerite trace to nil. Vanadium is present in most varieties of petroleum and asphalt,[7] and has also been reported in Argentine grahamite[8] and Peruvian impsonite.[9] Vanadium occurs in the more asphaltic crudes, but is not present in any quantity in non-asphaltic (i.e., paraffinaceous) crudes. A high vanadium content is generally associated with a high nickel content.[10] Uranium has similarly been reported in glance pitch found in Utah,[11] and molybdenum in asphaltic petroleum[12] and Holzheim (Swabian) oil shales.

(C) COMPOSITION OF ASSOCIATED NON-MINERAL CONSTITUENTS

Native asphalts often contain non-mineral impurities in the form of decayed vegetable substances of peat-like nature, which were originally present in the soil, now associated with the asphalt. These substances are derivatives of humic, or ulmic, crenic, etc., acids.

Certain tars and pitches as well as residual asphalts, which have been overheated in their process of manufacture, will carry variable amounts of so-called "free carbon." This in reality consists of hydrocarbon derivatives, polymerized under the influence of heat to an insoluble modification, similar in certain respects to bituminous coal. It is probable that the "free carbon" may under certain conditions consist in part of amorphous carbon, similar to lampblack. R. Hodurek has demonstrated that tars inherently contain certain insoluble constituents which may be removed by filtration (which he designates "Carbon I") and that other substances ("Carbon II") are formed by precipitation upon dissolving the tar in solvents, the quantity being dependent upon the particular solvent employed.[14]

(D) BEHAVIOR WITH SOLVENTS

This question will be taken up in detail in Chapter XXVI.

(E) BEHAVIOR ON SUBJECTING TO HEAT

On heating bitumens, the following reactions take place: at low temperatures they commence to distil without decomposition. Continued heating causes a rapid change in penetration and viscosity during the first few days or weeks, whereas the flash-point is least affected. The fusing-point changes regularly but markedly. There is a general decrease in acidity (i.e., acid value) which finally attains a constant figure.[15] As the temperature increases, a certain amount of cracking takes place, resulting in the depolymerization of both the distillate and residue. At still higher temperatures, the distillate continues to deploymerize, whereas the residue commences to polymerize with the formation of asphalt-like bodies. Beyond this, the residue again undergoes depolymerization with the evolution of a certain amount of fixed gases, likewise the formation of carbenes, free carbon, and eventually elemental carbon. Thus in the case of non-asphaltic and semi-asphaltic petroleums, polymerization takes place when the residue in the still reaches 600 to 800° F., whereby asphalt-like substances are produced. In other words the percentage of asphalt in petroleum is actually increased under these conditions. Beyond this temperature, cracking takes place, in which the asphalt is destructively distilled and decomposed into simpler molecules, yielding gases, liquid distillates and a residue of coke.[16]

The asphaltic pyrobitumens behave differently. When heated to between 600 and 800° F., they undergo "cracking." This is in reality a form of depolymerization in which complex molecules are broken down into simpler ones. As a result, the original substance, which is practically insoluble in organic solvents, increases materially in solubility. Elaterite and wurtzilite depolymerize and become completely soluble in benzol and carbon disulfide. Albertite is more difficult to depolymerize than elaterite or wurtzilite, requiring a higher temperature and an increased time of treatment. On the other hand, impsonite depolymerizes only slightly under these conditions. If heated to higher temperatures, the asphaltic pyrobitumens suffer destructive distillation, leaving a residue of coke. The depolymerization is similar to the action which takes place on melt-

ing fossil resins, such as copal, amber, etc., in manufacturing varnish.

A schematic outline of what occurs upon subjecting bitumens and pyrobitumens to heat is given in the following tabulation:

TABLE IV

BEHAVIOR ON SUBJECTING TO HEAT

		Heated under 300° C.	Heated 300–450° C.	Heated 450–700° C.	Heated 700–1500° C.
Bitumens.....	Non-asphaltic petroleums	Distil and the residues fuse	Residues depolymerize slightly	.....	.....
	Semi-asphaltic and asphaltic petroleums	Distil and the residues fuse	Residues polymerize, forming asphalt-like bodies	Depolymerize, yielding as distillate mostly open-chain hydrocarbons and a residue of coke	.....
	Mineral waxes	Small amount distils with slight decomposition, and the residues fuse	Depolymerize and distil		
	Asphalts and Asphaltites	Fuse	Distil more or less		
Pyrobitumens..	Asphaltic pyrobitumens	Infusible and insoluble	Depolymerize and become more soluble		
	Non-asphaltic pyrobitumens	Infusible	Unaffected	Depolymerize slightly and distil	Depolymerize, yielding as distillate, mostly cyclic hydrocarbons and a residue of coke

F. J. Nellensteyn has proposed the following theory to account for the formation of asphalts and the like:

(*I*) *Formation of Asphalts:*

$$\text{Hydrocarbons} \begin{Bmatrix} \text{oxidized} \\ \text{distilled} \end{Bmatrix} = \frac{\text{Transformed to carbon} + \text{Adsorbed protective hydrocarbon bodies}}{\text{Asphaltenes}}$$

(*II*) *Behavior of the Asphaltenes:*

$$\text{Asphaltenes} \begin{Bmatrix} \text{extracted} \\ \text{oxidized} \\ \text{heated} \end{Bmatrix} =$$

$$\frac{\text{Carbon} + \text{Small amount of adsorbed protective hydrocarbon bodies}}{\text{Free carbon}}$$

$$\text{Free carbon} \left\{\text{heated}\right\} = \frac{\text{Carbon} + \text{Very small amount of adsorbed hydrocarbon bodies}}{\text{Retort carbon}}$$

Resulting in a further decomposition of the protective bodies.

(*III*) *Synthesis of Asphaltenes:*

$$\text{Retort carbon} \left\{\text{electrically atomized in the presence of protective hydrocarbon bodies}\right\} = \frac{\text{Carbon} + \text{Adsorbed protective hydrocarbons}}{\text{Asphaltenes}}$$

(F) REACTIONS WITH GASES

(a) Oxygenation. On blowing air through bituminous substances at fairly high temperatures, hydrogen is removed in the form of water, and at the same time the hydrocarbons polymerize, forming bodies of higher molecular weight and more complex structure. The reaction may be roughly represented as follows:

$$C_xH_y + O = C_xH_{y-2} + H_2O$$

Analysis shows that little or no oxygen actually combines with the asphalt.[17] One theory is that polyhydric compounds acidic in character, are formed during the intermediate stage of oxidation, which upon further heating change to anhydrides similar to the polynaphthenic acids, with progressive condensation and polymerization. Various bituminous substances are affected differently by the blowing process. Asphalts present in petroleum are readily converted into tough, rubber-like products having a higher fusing-point and much more resistant to temperature changes. Fatty-acid pitch behaves in a similar manner. Natural asphalts are affected very much less, and pitches derived from coal and wood are scarcely affected at all. Even the petroleum asphalts themselves are affected differently, depending upon their origin and chemical characteristics. This subject will be gone into more fully under the heading "Petroleum Asphalts."

(b) Hydrogenation. This is carried out by heating bituminous substances (e.g., coal, asphalt, paraffin wax, tars, etc.) with hydro-

gen in an autoclave at high pressures (100 to 200 atmospheres) at 400 to 450° C. The process was discovered by Friedrich Bergius,[18] and has been termed "berginization." The following changes take place:

(1) Thermal decomposition, i.e., cracking.

(2) Hydrogenation, whereby the unsaturated hydrocarbons resulting from the thermal decomposition, at their moment of formation, react with the hydrogen, producing saturated hydrocarbons of lower boiling-points.

The process differs from straight thermal decomposition in the following respects: extensive polymerization is avoided and the deposition of carbonaceous matter is almost entirely prevented, moreover, as the reaction proceeds the pressure in the autoclave falls, due to the absorption of hydrogen, whereas in the absence of hydrogen, the pressure would increase due to the formation of fixed gases. Finally, the end product consists largely of saturated hydrocarbons, whereas in the cracking processes, unsaturated bodies will predominate. The resulting products are characterized by a higher hydrogen content. Any oxygen present in the original material is converted into water, and any nitrogen into ammonia.

In treating petroleum by the Bergius hydrogenation process, hydrogen gas is introduced at a pressure of about 3600 lbs. per sq. in. in the presence of a catalyst. Molybdic anhydride and sulfur[19] seems to be most favored, although numerous other catalyzers have been proposed, including chromium, tungsten, uranium, manganese, cobalt, etc., compounds, also metallic iron or nickel.[20] The catalyst should be sulfur-resistant and serve to speed up the reaction and eliminate the oxygen from the hydrogenated product. Relatively impure hydrogen, containing hydrogen sulfide, is used, produced from coal or coke by the water-gas process.

Asphaltic crudes of all characters (e.g., Venezuelan, Panuco, Colombian, etc.), likewise residues from refinery crudes, as well as cracking-plant tars, may be converted into volatile distillates, free from asphalt and low in sulfur, with volumetric yields somewhat in excess of 100% (although the specific gravity of the product is usually less than the crude stock). Dark asphaltic constituents are converted into practically colorless hydrocarbons of the gasoline range. In other words, the hydrogen will combine with the colloidal

carbon present in the asphalt present. In actual practice, the petroleum is first topped to remove all the gasoline present. From mixed-base crudes, highly paraffinic products may be produced, such as gas-oils, burning oils and high-grade lubricants. From paraffin crudes, so-called aromatic products may be produced, as for example, a highly anti-knock gasoline. No coke is produced in the process.

The charging stock, together with sufficient hydrogen, is pumped through heat-exchangers to a coil-furnace, and thence into the reaction vessel containing the catalyst where it is subjected to the required temperature and pressure. Units have been installed capable of treating 5000 barrels of crude daily.[21] The final products with the gases pass through heat-exchangers and coolers to a high-pressure separator, where the liquid reaction products are separated from the unconsumed hydrogen and other gases, which latter are scrubbed with oil under pressure to remove the hydrogen sulfide, and the purified gas used over again.

The following five adaptations of hydrogenation appear to be of most importance in petroleum refining:[22]

TABLE V

Process	Charging Stock	Primary Result	Approximate Volumetric Yield of				Total Volumetric Yield	Gas Formation
			Gasoline	Burning Oil	Gas Oil	Lube Oil		
			%	%	%	%	%	% *by wt.*
1	High-sulfur, asphaltic heavy residue	Asphalt and sulfur elimination with simultaneous conversion entire charge into distillate oils	30	....	71	...	101	2 to 3
2	Low-grade lube distillate	Production premium lube, particularly as regards temperature-viscosity relationship, flash, carbon, and gravity	10	....	29	65	104	0.5 to 1.5
3	Low-grade burning oil distillate	Production of low-sulfur premium grade burning oil	30	73	...	...	103	0.5 to 2
4	Cracked naphtha	Desulfurization and gum stabilization without deterioration in yield and knock rating characteristic of acid treating	100	....	...	...	100	0.5
5	Paraffinic gas oil	Production of low-sulfur, low-gum, good antiknock gasoline	65 to 100	....	...	...	70 to 100	5 to 35

If the charging stock is paraffinic in character, it is heated to 750° F., and if aromatic, to 1000° F. The reaction is exothermic.[23] It is advocated that very heavy asphaltic crudes be treated in three

stages, at pressures of 1000, 200 and 20 atmospheres, respectively.[24] The residual product obtained from the hydrogenation of residual asphalts has characteristics somewhat similar to those of blown petroleum asphalt.[25] Partial hydrogenation of petroleum stocks at 900 to 950° F., without using a catalyst, is claimed to result in the precipitation of any asphalt present.[26] Low-temperature coal-tar (topped to 200° C.), when hydrogenated with a mixture of molybdic acid and sulfur as catalyst, under an initial pressure of 145 atmospheres, yielded 99.2% of hydrogenated oil. The reaction started at 200° C., which increases to a maximum of 360° C.[27] The product is free from tar acids and pitchy matters.[28] Coke-oven tar showed an increase in volatile products below 300° C. from 31.7 to 65.5%, and in this case the hydrogen was absorbed by the liquid residue as well as by the fixed gases. Paraffin wax was entirely converted into liquid and gaseous products. Similarly, Alberta sand-asphalt has been hydrogenated under a pressure of 1470 lbs. at 380 to 410° C. in the presence of ammonium molybdate and aluminum chloride, or $NiCO_3$ and Fe_2O_3 or CaO (to remove the sulfur), being converted into 80 to 90% by weight of oils based on the asphalt present in the raw material, and involving the absorption of 3% by weight of hydrogen.[29]

A modification of the preceding has been devised by Meilach Melamid, which consists in atomizing asphaltic petroleum into a heated reaction chamber together with hydrogen, so that the two are brought in contact with a catalyzer (e.g., tin or bismuth alloys melting below 700° F.).[30] In this process the hydrogenation and continuous distillation of the crude oil take place simultaneously. In the case of a Panuco (Mexican) crude, the distillate to 320° C. was increased from 19 to 26 per cent, and in the case of a brown-coal tar resulting from the Mond gas process, the pitch residue was reduced from 46.8 per cent to 0.0 per cent, with a corresponding increase in the percentages of distillates.

(G) REACTIONS WITH ACIDS

(a) Liquid Sulfur Dioxide. This agent has the property of dissolving unsaturated hydrocarbons at low temperatures (— 7° to — 11° F.), whereas the saturated hydrocarbons remain substan-

tially insoluble. Advantage is taken of this fact in certain commercial refining operations, also in the laboratory examination of bituminous substances.[31] In general, the higher boiling-point paraffins, naphthenes and naphthenic acids (i.e., over 175° C.) are insoluble, whereas the aromatics and the true unsaturated hydrocarbons (containing two or more double bonds) are miscible with liquid sulfur dioxide in all proportions. Sulfur and its organic combinations, nitrogen compounds, resinous and asphaltic substances are also soluble, though their solubilities decrease with increasing complexity of the molecule.[32]

(b) Sulfuric Acid and Sulfur Trioxide. Concentrated sulfuric acid, or a mixture of concentrated sulfuric acid with sulfur trioxide reacts with bituminous substances in a somewhat complicated manner, involving both the formation of sulfo derivatives and that of simple solution, accompanied by a certain amount of polymerization. In general, it may be stated that the following classes of substances are removed by concentrated sulfuric acid: unsaturated and aromatic hydrocarbons, asphaltic bodies, saponifiable constituents and alkaline bases. This reaction forms the basis of the method used commercially for refining petroleum distillates and is likewise used in the laboratory under accurately controlled conditions for the separation of saturated and unsaturated hydrocarbons. (See Test 34.) A process has been described which consists in heating residual asphalt with 20 to 25 per cent of concentrated sulfuric acid to 120° C. which is then gradually increased to 240° C. The addition compounds are separated and the residue (i.e., sludge asphalt) used for molding purposes.[33]

(c) Nitric Acid. Strong nitric acid reacts in two ways, namely in the formation of nitro derivatives and in the oxidation of the hydrocarbons. Which of these predominates depends upon the nature of the hydrocarbons, the strength of the acid, the temperature, etc. Although much work has been done in investigating the character of end-products, but little commercial use has been made of this reaction.[34] Similar products are obtained by heating tars, pitches or asphalt with substances containing NO_2 or NO_3 radicals.[35] It has been noted that certain of the nitro addition products are soluble in acetone and alcohol, thus differing from the hydrocarbons from which they were derived.

(d) **Sulfuric Acid and Formaldehyde.** Formaldehyde in the presence of concentrated sulfuric acid reacts with unsaturated cyclic hydrocarbons and asphaltic constituents with the formation of an insoluble formolite, whereas the other classes of bodies remain unaffected. This reaction is utilized in the laboratory examination of bituminous substances. (See Test 35.) It has also been proposed to incorporate the formolite with asphalts, pitches, etc., to produce molded products, electrical insulation, etc.[36]

(e) **Phosphoric Acid.** Phosphoric acid has been proposed for refining mineral oils, yielding "sludge asphalt." [37]

(H) REACTION WITH ALKALIES

Strong alkalies react with the saponifiable constituents present, including the following groups of substances: free asphaltous acids, asphaltous acid anhydrides, fatty and resinous constituents. The saponifiable constituents in commercial bituminous substances vary from a trace in the case of bitumens and asphaltic pyrobitumens, to as high as 90 per cent and over in the case of rosin pitch and fatty-acid pitch. The effects of alkalies will be considered later in greater detail.

(I) REACTIONS WITH METALLOIDS

(a) **Sulfur and Sulfur Dichloride.** Under the influence of heat, sulfur has the same condensing effect upon bituminous substances as oxygen, since it results in the removal of hydrogen, but in this instance in the form of gaseous hydrogen sulfide. The reaction may roughly be represented as follows:

$$C_xH_y + S = C_xH_{y-2} + H_2S$$

A certain amount of polymerization also takes place, and where the sulfur is used in excess, it is likely that a certain amount remains in combination with the bituminous substances in the form of addition products. The process has been termed "vulcanization," and is used to improve the stability of bituminous mixtures. This will be considered more fully under the description of the respective substances in subsequent chapters.

Sulfur dichloride combines with bituminous substances under

moderate heat, with the liberation of hydrochloric acid as a product of the reaction, which may be roughly indicated by the following:

$$C_xH_y + S_2Cl_2 = C_xH_{y-6} + 2H_2S + 2HCl$$

This reaction is merely of academic interest.[38]

(b) **Selenium.** Selenium combines with asphalts, etc., at 160 to 300° C. in a manner analogous to sulfur, with the liberation of hydrogen selenide, a proportion of the selenium remaining in combination with the residue.[39]

(c) **Phosphorus.** It has been noted that phosphorus, or P_2O_5 under the influence of moderate heat, will cause asphalts to polymerize, and in part combine chemically with the asphalt, resulting in an increase of hardness, fusing-point and stability.[40]

(d) **Halogens.** Chlorine and iodine [41] eliminate hydrogen and result in the deposition of carbon, in accordance with the following reactions:

$$C_xH_y + Cl = C_xH_{y-1} + HCl$$
$$C_xH_y + Cl = C_{x-1}H_{y-1} + C + HCl$$

Similar reactions take place with iodine, also boron fluoride.[42] The relation between the "iodine number" of asphalts and their structure has also been investigated.[43]

Halogens seem to destroy the protective colloids that keep the colloidal carbon in suspension and result in the deposition of elemental carbon, if the reaction progresses far enough. Upon treating asphalt with chlorine gas in the presence of a trace of iodine at 200 to 250° C., the fusing-point is increased materially, and a hard, glossy product is obtained having a glossy fracture.[44] Similarly, a soft coal-tar pitch will have its fusing-point increased from 25 or 30° F. to 120° F.,[45] likewise its adhesiveness increased.[46] It is claimed that hexachlorethane will react in a similar manner.[47] A process has also been proposed which involves treatment with chlorine below 130° C. in the presence of antimony chloride, phosphorus chloride, or iodine; [48] likewise a method of treating with concentrated sulfuric acid, followed by $FeCl_3$, $AlCl_3$, or PCl_5.[49]

(J) REACTIONS WITH METALLIC SALTS

Bituminous substances act as "dispersoids," in that they have the property of dispersing colloidal solids, including certain metallic salts. Trinidad asphalt is a typical example, in which the asphalt acts as a dispersoid for adventitious mineral matter of colloidal nature, consisting principally of colloidal clay. That the clay is present in a colloidal state is evidenced by the following facts: (1) a dilute solution of Trinidad asphalt in carbon disulfide, upon standing, retains indefinitely the clay in suspension, and moreover it cannot be separated upon centrifuging, or upon passing the solution through an ultra-filter; and (2) an examination of the solution through the ultra-microscope shows the mineral particles to be in a characteristic state of active motion, known as Brownian movement or "pedesis," which may be made to cease under the influence of an electric field ("cataphoresis"). This subject will be discussed further in Chapter XXIV under the heading "Colloidal Particles—Liberated 'In Situ'." Other metallic salts which will disperse in bituminous substances include sulfates and selenates, chlorides, oxides, acetates and borates.

CHAPTER IV

GEOLOGY AND ORIGIN OF BITUMENS AND PYROBITUMENS

GEOLOGY

Age of the Geological Formations. The earth's crust has been divided into natural groups or strata in the order of their antiquity. There are five main divisions, which range in sequence as follows:

1. Quaternary or Post-tertiary, representing the strata now in the process of formation.
2. Tertiary or Caenozoic, embracing the age of recent life.
3. Secondary or Mesozoic, representing the less recent life.
4. Primary or Paleozoic, representing the so-called "ancient life."
5. Archaean or Azoic, representing the so-called lifeless strata.

These divisions are recognized by the distinctive organic remains, fossils, minerals and other characteristics. They are classified into various "systems" as shown in Table VI.

The systems form a chronological time-chart indicating the relative ages of the earth's strata. The systems are further sub-divided into groups which differ in different localities, but it will be unnecessary to consider their sub-classification here.

Petroleum occurs in all of the geological horizons from the Recent down to the Pre-Cambrian. Certain systems are richer than others, especially the Pliocene, Miocene, Oligocene, Eocene, Carboniferous, Upper Devonian and Lower Silurian (Ordovician). Asphalts, asphaltites and non-asphaltic pyrobitumens are found in all the systems from the Pliocene to the Silurian. Mineral waxes are found largely in the Pliocene, Miocene, Oligocene, Eocene and Cretaceous.

The non-asphaltic pyrobitumens do not occur in the older Paleozoic formations i.e., the Silurian or Cambrian Systems). The Carboniferous System contains the most valuable coal deposits; the Permian and Triassic Systems contain coals of inferior quality, and

the coals found in the Jurassic, Cretaceous, Eocene and Oligocene Series are still more inferior in quality. Lignite occurs in the Oligocene and Miocene Series, and peat in the Pliocene and Pleistocene. The Pre-Cambrian Series consists largely of crystalline, metamorphic rocks of volcanic and igneous origin. The non-asphaltic pyrobitumens, as might be expected, do not occur in rocks of this character. Graphite, however, occurs in the Pre-Cambrian rocks, and may possibly have been derived from vegetable matter, although no signs of associated plant remains have been found in these rocks.

TABLE VI

Era	System	
Quaternary or Post-tertiary	Recent or Post-glacial	Historic, Prehistoric, Neolithic
	Pleistocene or Glacial	
Tertiary or Caenozoic	Pliocene	
	Miocene	
	Oligocene	
	Eocene	
Secondary or Mesozoic	Cretaceous	Upper, Middle, Lower
	Jurassic	Upper, Middle, Lower
	Triassic	
Primary or Paleozoic	Permian	
	Carboniferous	Upper, Lower
	Devonian	Upper, Middle, Lower
	Silurian	Upper, Lower (Ordovician)
	Cambrian	Upper, Middle, Lower
Archaean or Azoic	Pre-Cambrian	

The particular geological system is of value in enabling us to prospect and trace deposits of bitumens and pyrobitumens in any given locality.

Character of the Associated Minerals. Bitumens and pyrobitumens, with but few exceptions, are found in sedimentary deposits of sand, sandstone, limestone and sometimes in shale and clay. Rare

occurrences have been reported in igneous rocks, but then only in very small quantities.

Modes of Occurrence. Bitumens and pyrobitumens are found in nature in the following ways:

1. Overflows:
   - (*a*) Springs.
   - (*b*) Lakes.
   - (*c*) Seepages.
2. Impregnating Rocks:
   - (*a*) Subterranean pools or reservoirs.
   - (*b*) Horizontal rock strata.
   - (*c*) Vertical rock strata.
3. Filling Veins:
   - (*a*) Caused by vertical cleavage.
   - (*b*) Caused by upturning.
   - (*c*) Caused by sliding.
   - (*d*) Formed by sedimentation.

Springs. Petroleum and the liquid forms of asphalt only are found in springs (Fig. 25). These emanate from a fissure, crevice, or fault which permits the petroleum or liquid asphalt to rise to the surface from some depth. Petroleum or asphalt springs have been reported in various parts of the world, but are rarely of commercial importance.

Lakes. Asphalt only is sometimes found in lakes, which are in reality springs on a very large scale (Fig. 26). Some of the largest and most valuable deposits occur in this form, the best known being the lakes at Trinidad and Venezuela. It is probable that the asphalt is forced up from below in a liquid or semi-liquid condition by the pressure of the oil and gas underneath, which causes it to flow through a fissure or fault and spread over a large area at the surface. Lake asphalts are moderately soft where they emanate, but harden on exposure to the elements.

Seepages. These occur in the case of petroleum or liquid asphalts, and usually from cliffs or mountains bearing impregnated rock. Either the pressure of the material itself or the heat of the sun causes a certain quantity, usually not very large, to flow out of the rock and run towards the lower level (Fig. 27). Seepages are often found where a rapidly flowing stream of water cuts its way

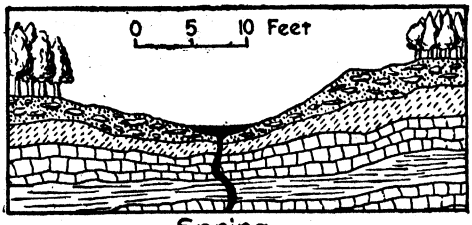


FIG. 25

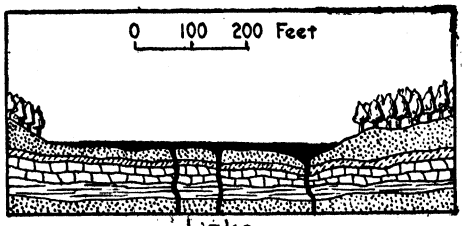


FIG. 26

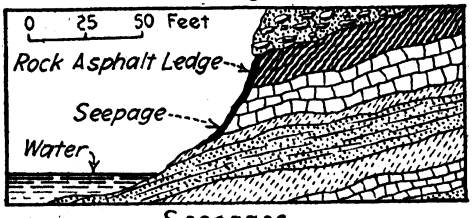


FIG. 27

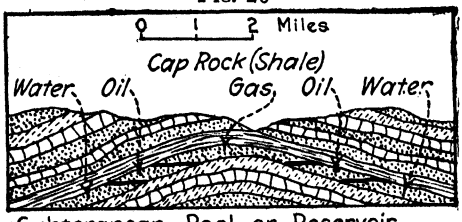


FIG. 28

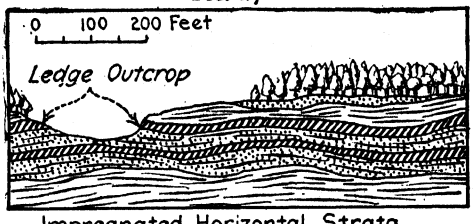


FIG. 29

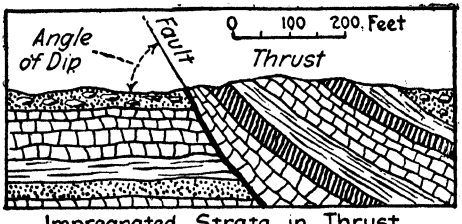


FIG. 30

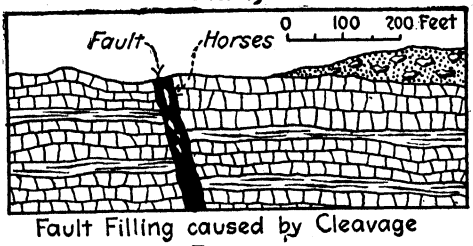


FIG. 31

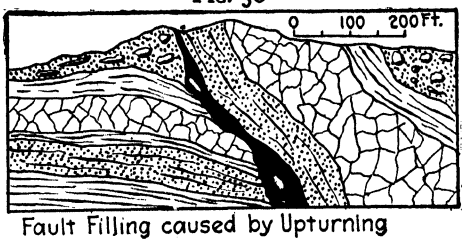


FIG. 32

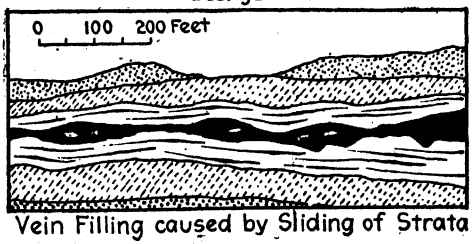


FIG. 33

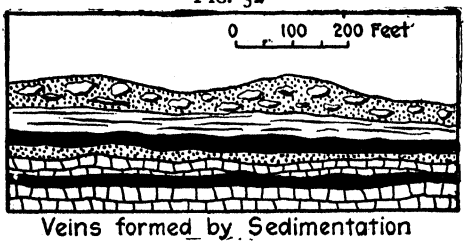
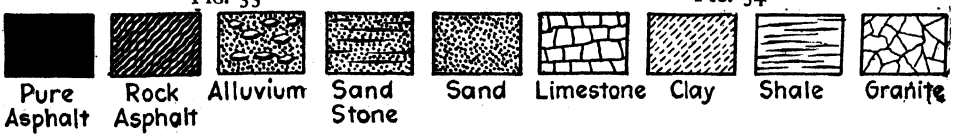


FIG. 34



FIGS. 25–34.

through strata of rock impregnated with petroleum or asphalt. From a commercial standpoint, seepages of asphalt or petroleum are of little value.

Subterranean Pools or Reservoirs. Practically all deposits of petroleum of any magnitude occur below the surface of the earth in subterranean "pools" or "reservoirs." These consist of porous sand, sandstone, limestone (or dolomite) with a more or less impervious rock cover. The porous bed is exemplified by coarse-grained sandstone, conglomerate, or limestone. The limestone may have been dense as it existed originally, but rendered porous in the course of time by conversion into dolomite, with the consequent production of voids due to shrinkage, since dolomite occupies less space than the original limestone. The petroleum is carried in the interstices of the porous rock and prevented from volatilizing or escaping by an impervious cover known as the "cap-rock," usually composed of shale or a dense limestone. The main supplies of petroleum have been obtained from regions which have been comparatively undisturbed by terrestrial movements. In such cases the accumulation of petroleum underneath forms what is known as a "pool" or "reservoir" (Fig. 28).

Impregnated Rock in Strata. Liquid to semi-liquid asphalts occur in this manner. Rocks impregnated with asphalt are produced in two ways, viz.:

(1) By the gradual evaporation and hardening of an asphaltic petroleum due perhaps to the disturbing or removal of the cap-rock, leaving the asphalt residue filling the interstices of the stratum carrying it. These are usually found in horizontal strata (Fig. 29).

(2) By the liquid asphalt being forced upward under pressure, or drawn upward by capillarity from underlying strata into a porous rock layer above it. These are usually found in the region of a "thrust" or upturning of the earth's strata (Fig. 30).

Filling Veins. The harder asphalts, asphaltites and asphaltic pyrobitumens are most commonly found filling fissures in a more or less vertical direction, caused by "faulting." In geology, a "fault" is a more or less vertical crack in the earth's surface brought about by the contraction and uneven settling of the strata. This is occasioned by a greater movement in the rock on one side of the fault's plane than on the other, as illustrated in Fig. 31, or by the upturning

of a section of the earth's crust as shown in Fig. 32. Faults allow the liquid or molten asphalt to force its way up from underneath and fill the crevice. As will be described later, after the asphalt hardens in the fault, it might in time become metamorphosized and converted into an asphaltite or asphaltic pyrobitumen. Non-asphaltic pyrobitumens are never found in faults, probably because they are incapable of softening or melting under the action of heat, either in their original state or afterwards.

Sometimes we find the harder asphalts, asphaltites and asphaltic pyrobitumens filling a more or less horizontal fissure or cleavage crack, brought about by the sliding of two strata, one upon the other. The opening between the strata becomes filled with the liquid or melted asphalt, forced up under pressure through a crevice from below, which then hardens, giving rise to a horizontal vein as shown in Fig. 33.

Horizontal veins are sometimes derived from prehistoric asphalt "lakes," perhaps similar to our present Trinidad or Venezuela lakes. In time, these harden, and become submerged in water, due perhaps to a movement in the level of the earth's crust. The water permits the sedimentation of mineral matter, so that the lake is gradually converted into a horizontal vein. If the liquid asphalt again breaks through a fault or fissure, it will form a superimposed vein, or perhaps a series of such veins between sediments, as shown in Fig. 34.

In the case of non-asphaltic pyrobitumens, the veins were unquestionably formed by a process of sedimentation. The vegetation from which these were derived, originally grew in swampy or marshy localities, presumably about the mouths of rivers. As the vegetation died, it became covered with sediments of sand or clay carried down by the water, or by calcium carbonate precipitated from the water, which in turn formed soil for subsequent growths. These gave rise to the future veins of non-asphaltic pyrobitumens, which are similar structurally to the preceding (Fig. 34).

Movement of Bitumens in the Earth's Strata. It is a singular fact that petroleum, mineral waxes, asphalts and asphaltites are not always found in the same locality in which they originated. They have the power of migrating from place to place, and many deposits are still in the process of migration. A "primary deposit" is one in which the bituminous material is still associated with the same rocks

in which it originated. A "secondary deposit" is one to which the material has subsequently migrated. Bitumens usually migrate while in a liquid or melted condition, although in certain rare instances the migration has been induced by the action of flowing water while the bitumen is in the solid state.

The main causes for the movement of native bituminous substances in the earth's surface are as follows:

(1) *Hydrostatic Pressure.* This is largely responsible for the accumulation of petroleum in pools or reservoirs. At some distance below the earth's surface there is an accumulation of ground water, the level of which varies in different localities and during different seasons. The petroleum, being lighter than the water, floats on its surface. As the level of the ground water varies, it will move the petroleum about through interstices in the rock. The water tends to push the oil ahead of it, and this will account for the accumulation of the petroleum in the form of pools or reservoirs underneath a cap of a dense and non-porous strata through which it cannot permeate. This will also explain why there is often an accumulation of petroleum in the ground near the top of a hill or mountain. Oil and gas are often encountered under pressure, due to the hydrostatic head of water.

Hydrostatic pressure may also cause the migration of solid asphalts, as for example in the case of the Dead Sea, where masses become detached from the bottom and are caused to float upward by the higher gravity of the water, due to the large percentage of salt dissolved in it.

(2) *Gas Pressure.* It is probable that the action of the heat or other forces below the surface of the earth, tend partially to vaporize certain bitumens, so that the resulting gas will force them into the overlying strata near the surface. In other instances the effect of faulting, crumpling, upturning, erosion and other movements of the earth's strata exposes the oil- or asphalt-bearing formations, and enables the gas pressure to force them to the surface. Natural gas exists under great pressure in certain localities. Many gas wells in the Baku and Pennsylvania fields have registered a pressure of 600 to 800 lb., and even as high as 1000 lb. per square inch. This may be accounted for by the fact that as the gas is constantly being generated, it accumulates inside of the earth's surface and has no access to escape owing to the density of the strata above.

(3) *Capillary.* This force takes place in dry porous rocks and acts on permanently liquid bitumens, or bitumens solid at ordinary temperatures but transformed to the melted state by the action of heat. Under these conditions the bitumen will soak into the

pores of the rock or sand and gradually fill the interstices. Capillarity is a very much stronger force than gravity, although other forces, such as the action of heat (see under 5) may be partly responsible. The finer the pores in the rock, the greater will be the capillary force. Rocks saturated with moisture tend to resist the action of capillarity, which is most effective in the dry state.

(4) *Gravitation.* The natural weight of the overlying strata caused by gravitation sets up a pressure where there are accumulations of petroleum or other forms of liquid bitumen underneath, and if a fissure or fault occurs in the earth's crust, the bitumen, being softer than the surrounding rock, will be forced to the surface. Gravitation is therefore selective in its action and by exerting a greater pull on the heavier bodies will tend to force the lighter ones upward. Under other circumstances, where the substances are not confined, the result of gravitation is to cause the petroleum or liquid asphalt to ooze from the overlying rock matrix in the form of "seepages."

(5) *Effect of Heat.* Heat is also a large factor in causing the migration of bituminous substances. Its effect is variable. Under certain circumstances it will convert the solid bitumens into a liquid state and thus enable them to be acted upon by the various forces considered previously. Under other conditions, heat in the interior of the earth will vaporize the bitumens such as petroleum and force them upward. Again, if the heat is sufficiently intense, it is apt to cause the bitumens to undergo destructive distillation, the distillate condensing in the upper and cooler layers.

ORIGIN AND METAMORPHOSIS OF BITUMENS AND ASPHALTIC PYROBITUMENS

Probable Origin of Bitumens and Asphaltic Pyrobitumens. Although much has been written on this subject, no generally acceptable conclusions have been reached. The discussion has in the main centered about the origin of petroleum, as this is conceded to be the mother substance, from which the other bitumens and pyrobitumens are supposed to have been produced by a process of metamorphosis. The theories have been divided into two classes, namely, the inorganic and the organic. We will consider these in greater detail.

Inorganic Theories. It is contended that the interior of the earth contains free alkaline metals, presumably in a melted condition. These at high temperatures would react with carbon dioxide, forming acetylides which in turn produce hydrocarbons of the acetylene series upon coming in contact with water. The acetylenes being

unsaturated would have a tendency to combine with free hydrogen and give rise to the olefine and paraffin series.

Still another theory based on similar lines assumes the presence of metal carbides, including iron carbide, some distance below the surface of the earth. These are supposed to decompose on coming in contact with water and produce hydrocarbons, which upon condensing in the cooler upper strata give rise to petroleum. This, however, is mere speculation, for no iron carbide has ever been found. The occurrence of hydrocarbon gases in volcanic emanations has been cited to substantiate this theory.

The cosmical hypothesis is based upon the assumption that hydrocarbons were present in the atmosphere which originally surrounded the earth, after it had been thrown off by the sun. These hydrocarbons are claimed to have been formed by a direct combination of the elements carbon and hydrogen in the cosmic mass. As the earth cooled, the hydrocarbons condensed in the earth's crust, giving rise to deposits similar to those existing to-day. This theory has also been connected with the carbide theories, upon the assumption that at the high temperatures to which the gases must have been subjected at the time they were thrown off by the sun, and before they condensed, the first compounds formed were carbides, silicides, nitrides, and the like. As oxidation would not commence until some time later, it is assumed that these carbides would remain locked up in the interior of the earth for geologic ages, and then gradually give rise to hydrocarbons upon being decomposed through the agency of water.

Vegetable Theories. It has long been known that certain hydrocarbons result during the decay of vegetation. The hydrocarbon methane (CH_4), otherwise known as "marsh gas" is produced in this manner, but only in comparatively small amounts. Similarly, methane has been detected in the gases resulting during the decay of seaweed.

It has been shown by others that under certain conditions hydrocarbons may be produced artificially by the fermentation and decay of certain forms of cellulose, including woody fiber. Still other scientists maintain that petroleum is produced by microscopic plants known as diatoms, which occur abundantly in peat beds, and certain bogs. These organisms are found to contain minute globules of

oily matter distributed in the plasma, and moreover, a waxy substance resembling ozokerite may be extracted by solvents from the diatomaceous peat. It is contended that this oil will in time and under pressure become converted into liquid petroleum, and at higher temperatures and pressures possibly into asphalt. In support of this hypothesis a bed of peat has been described near Stettin, Germany, consisting largely of diatoms and from which hydrocarbons have been extracted in quantities up to 4 per cent.

Another theory based on similar lines infers that petroleum is derived from a slimy substance rich in organic matter known as "sapropel," composed largely of algae, which accumulates at the bottom of stagnant waters. This slime becomes covered with sediments which through the agency of moisture, time and pressure, is assumed to give rise to petroleum, and under certain conditions, to asphalt. It is of interest to note that petroleum and asphalt have been produced in the laboratory by the hydrolysis of algae in acid solutions.[1]

In a similar manner, bitumens are claimed to have been formed from deposits of vegetable matter, including various marine plants, seaweeds, etc., which accumulate at the bottom of the ocean. Just as the non-asphaltic pyrobitumens (e.g., coal) are produced by the decomposition of terrestrial vegetation, it is contended that bitumens have arisen from the decay of marine plants. This theory has a number of adherents. The optical activity of certain petroleums has been cited to substantiate the contention, since oils derived from organic matter can only possess this property. It has been proven that hydrocarbons produced from inorganic substances, such as metal carbides, do not exhibit optical activity.

Still another theory, advocating the vegetable origin of petroleum, assumes its derivation from peat, lignite or coal, which have been subjected to a sufficiently high temperature to undergo a process of destructive distillation, resulting in the production of liquid and gaseous hydrocarbons. This is supposed to have occurred at great depths below the earth's surface and the hydrocarbons condensed in upper layers.

The asphaltic pyrobituminous shales are similarly claimed to have generated petroleum under the action of heat, based on the well-known fact that when these shales are distilled commercially,

petroleum-like oils are produced. It is contended that the shales themselves were derived from gelatinous algae whose remains are still recognizable in certain of them with the aid of a microscope.

Animal Theories. In a similar manner, petroleum and asphalt are supposed to have been produced from the accumulation of animal matter at the bottom of the ocean, which in time decomposed into hydrocarbons. The presence of nitrogen in all forms of bitumen is cited in substantiation of its production from albuminoid matter. The remains of molluscs and fish are present in certain asphaltic pyrobituminous shales, including the Albert series of New Brunswick, and in many rocks carrying petroleum and asphalt. Deposits of the latter have been reported in Galicia, Wyoming, and are particularly noticeable in the case of oil and asphalt deposits in Uvalde County, Texas, and southeastern California. In Egypt, shells are also found filled with bitumen. Others contend that the living cells are in some manner absorbed into the pores of coral reefs, and that these in time result in the formation of bituminous limestone.

Substances closely resembling petroleum or bitumens have been produced artificially by subjecting fish albumin to heat, under pressure. Asphalt-like substances have been produced by heating gelatine, casein, calcium carbonate and magnesium carbonate in an atmosphere of hydrogen at 240° C. under pressure of 33 atm. The solution of the resultant product in carbon disulfide exhibited Brownian movement and the particles were flocculated by ether.[2] Animal fats have similarly been converted into hydrocarbons boiling below 300° C. The conversion of fats and albuminous substances into petroleum is said to depend upon three factors, namely, pressure, temperature and time. The variations in the composition of petroleum found in different localities, are accounted for by variations in one or more of these factors.

In conclusion it might be stated that probably all three theories embody certain elements of truth. The cosmical hypothesis is sustained by the fact that hydrocarbons have often been found in meteorites, although this supposition has been refuted.[3] The inorganic theory is borne out by the fact that hydrocarbons occur in volcanic emanations. The vegetable and animal theories in turn are supported by the presence of bitumens and pyrobitumens in rocks of

sedimentary character, often carrying vegetable and animal fossil remains. Mabery contends that the presence of nitrogen in petroleum derived from all the principal oil fields, exists in the form of combinations which could only have had their origin in vegetable or animal remains.[4]

It is highly probable, therefore, that bitumens owe their origin to two or more of the theories which have been discussed, and which would account for their varying chemical composition and physical characteristics.

Metamorphosis of Mineral Waxes, Asphalts, Asphaltites and Asphaltic-Pyrobitumens from Petroleum. Although there seems to be a wide difference of opinion regarding the origin of petroleum authorities are pretty well agreed that petroleum when once formed, is gradually converted into the other types of bitumen and pyrobitumens, under the influence of time, heat and pressure. This process of transformation is known as "metamorphosis."

Several chemical processes are involved, e.g., oxidation, sulfurization, polymerization (i.e., the combination of like molecules) and condensation (i.e., the combination of unlike molecules). Some natural asphalts are derived through the slow evaporation of lower boiling-point fractions from the original petroleum; others indicate conversion by heat and pressure; still others show evidence of slow oxidation. It is likely that a combination of these three processes occur simultaneously. Asphalt associated with tar sands has undergone little change other than the loss of volatile fractions; on the other hand, gilsonite, glance pitch and grahamite appear to be products of reaction and conversion, rather than products of evaporation.

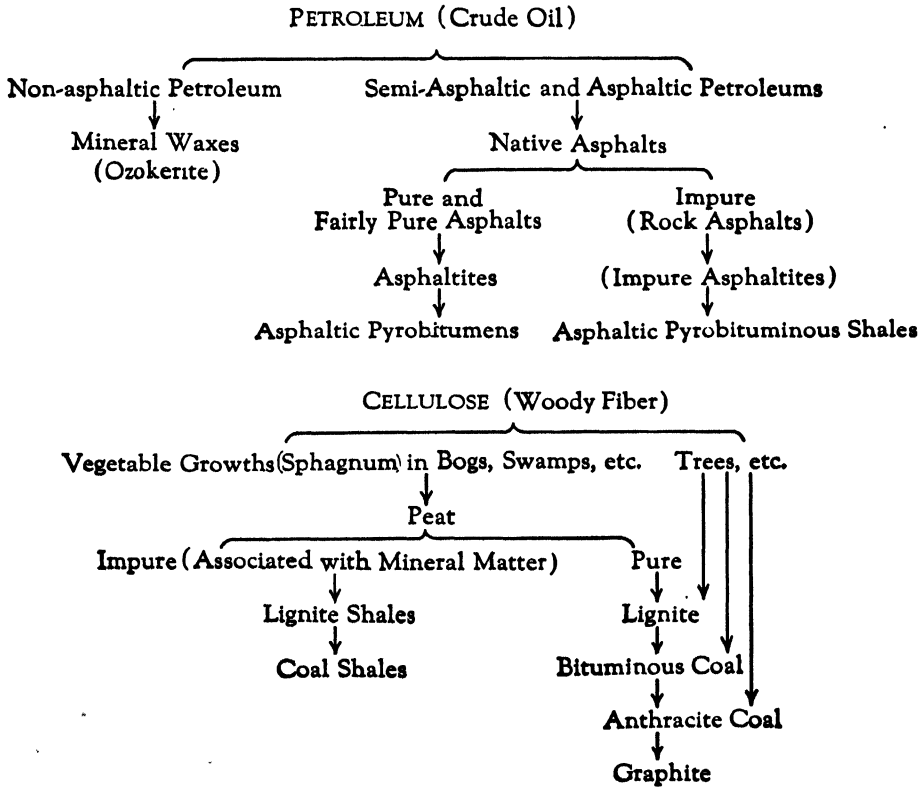
It is contended that mineral matter in a finely divided form, as for example "colloidal" clay, hastens the transformation of natural gas or petroleum, by acting as a catalyzer. This theory is advanced by Clifford Richardson.[5] In studying the well-known Trinidad asphalt lake, Richardson concludes that an asphaltic petroleum existing at a considerable depth is converted into a more solid form of bitumen, namely asphalt, upon being thoroughly emulsified with colloidal clay, sand and water through the medium of natural gas at a high pressure. During the metamorphosis, hydrogen is gradually eliminated, the hydrocarbons becoming enriched in carbon, and from a chemical standpoint more complex structurally. The changes

brought about during this process may be regarded as a form of polymerization, in which the hydrocarbon molecules become rearranged into more complex molecules of higher molecular weight.

The simplest hydrocarbons are present in petroleum. Those in mineral waxes are somewhat more complex, and both the structural complexity and the molecular weight increase in the case of asphalts and the asphaltic pyrobitumens. There are no sharp lines of demarcation between the various types of bitumens or asphaltic pyrobitumens. Each class gradually merges into another, and specimens will often be found on the border line, so that it is difficult to decide to which class they actually belong.

From this viewpoint we may regard petroleums as passing in gradual stages, under the influence of time, heat, pressure and catalyzers into the soft native asphalts, which in turn pass into harder native asphalts, and then into asphaltites and finally into the asphaltic pyrobitumens and asphaltic pyrobituminous shales.[6]

TABLE VII



It is highly probable that all deposits of asphalt are produced by metamorphosis from *asphaltic* petroleum. Similarly it seems likely that all deposits of mineral wax, such as ozokerite, etc., result from the metamorphosis of *paraffinaceous* petroleum.

Elaterite, wurtzilite, albertite, impsonite and the asphaltic pyrobituminous shales represent the final stages in the metamorphosis of petroleum. The first four are comparatively free from mineral matter. If the latter predominates, the product is known as an asphaltic pyrobituminous shale. The non-mineral matter contained in these shales has the same general characteristics as elaterite, wurtzilite, albertite or impsonite, depending upon how far the metamorphosis has progressed.

Table VII gives an approximate idea of the natural metamorphosis of bitumens and pyrobitumens, one from another.[7]

ORIGIN AND METAMORPHOSIS OF NON-ASPHALTIC PYROBITUMENS

The origin of non-asphaltic pyrobitumens has been definitely established. The associated fossil remains clearly prove that these have been derived from vegetable matter containing cellulose, a carbohydrate having the empirical formula $(C_6H_{10}O_5)_n$.

The decomposition of cellulose when the air is partly or wholly excluded, as would be the case when buried in the ground, results in the loss of carbon dioxide, methane, and water. In this manner, cellulose ultimately yields a series of products grouped under the heading of non-asphaltic pyrobitumens. The conditions favorable to their production seem to be the growth of vegetable substances about the mouths of rivers, combined with a change in water-level. The sediment carried down by the river, formed beds of sand or clay which sealed the vegetation in between the strata. In this manner the vegetable matter was protected from atmospheric oxidation and at the same time probably subjected to fermentative heat, also to a gradually increasing pressure, as successive layers accumulated. The vegetation doubtlessly embraced many different kinds, including trees, ferns, grasses, mosses, and the like. Fossil ferns are still clearly evident in coal beds. In other cases carbonized trees, roots and fibrous tissue are recognizable, and in still others, the resins originally present in the wood are found intact. Amber and fossil

copal often occur in peat, and large masses of vegetable resin have been identified in beds of lignite and bituminous coal.

Peat represents the first stage in the metamorphosis of coal from vegetable matter, and occurs in bogs or other swampy places. Very often on the surface of a bog or swamp we see the still living and growing plants. A little below, we find their decayed remains, and still deeper, a black glutinous substance saturated with moisture, known as "peat." [8]

The exact nature of the changes which take place in the transformation of vegetable matter into peat is not clearly understood. The ultimate analysis shows that the percentages of hydrogen and oxygen have diminished, and carbon correspondingly increased. In the most recent deposits, peat is loosely compacted, but as it accumulates under the sediments, it becomes compressed. A bed which was possibly once a foot thick might shrink to several inches. In all probability the pressure developed by the superimposed layers, aids in the transformation of peat into coal.

Lignite [9] or browncoal is intermediate between peat and bituminous coal. The most recent deposits approach peat in composition, and the oldest merge into bituminous coal. Lignite contains a larger percentage of carbon and smaller percentages of hydrogen and oxygen than peat. It is often associated with mineral resins or wax-like hydrocarbons * which may be extracted by means of solvents.

Jet, gagate, azabache and "black amber" are species of lignite.

Cannel coal [10] and bog-head coal and torbanite are in reality a sub-class of bituminous coal, rich in volatile matter. The former are supposed to have been derived from spores, spore cases, and resinous or waxy products of plants (e.g., sapropel). The absence of woody material gives cannel coal a uniform texture and grain not present in other coals, so that it breaks with a conchoidal fracture, and a splinter ignites in contact with a lighted match, burning like a candle, whence it derives its name.

* The associated resins or waxes, or asphalts, as they are termed by some, have been described under various names, including: Anthracoxenite, Bombiccite, Branchite, Butyrellite, Dinite, Dopplerite, Duxite, Dysodile, Euosmite, Fichtelite, Geomyricite, Hartine, Hartite, Hofmannite, Ionite, Köflachite, Leucopetrin, Leucopetrite, Melanchyme, Mellite, Middletonite, Muckite, Neudorfite, Neft-Gil, Phytocollite, Pianzite, Pyroretin, Refikite, Retinasphaltum, Retinite, Rochlederite, Schleretinite, Sieburgite, Trinkerite, Walchowite, Wheelerite, etc.

Bituminous coal falls between lignite and anthracite coal. It is often a matter of difficulty to determine where the lignites stop and the bituminous coals begin; similarly, the line of demarcation between bituminous and anthracite coals is not very distinct. Bituminous coals contain a larger percentage of carbon and smaller percentages of hydrogen and oxygen than lignite. The name "bituminous coal" is derived from the fact that this coal apparently softens and undergoes fusion at a temperature somewhat below that of actual combustion. The term, however, is a misnomer. The softening which takes place marks the point at which destructive distillation commences, accompanied by the formation of gaseous hydrocarbons. "Bitumen" does not actually exist in bituminous coal. That portion which dissolves in solvents (e.g., xylol, phenol, tetralin, etc.) has been termed "bituminic substance," since it resembles bitumens. The insoluble portion has been termed the "lignitic residue," since it resembles lignite. Bituminous coals contain a substantial portion of volatile matter, which causes them to burn more rapidly than anthracite, and with a larger amount of flame. The so-called "coking coal" is a class of bituminous coal, used in the manufacture of coke. The relationship between bitumens and coal has also been traced by the similarity of the substances separated by the selective action of certain solvents, in their deportment under heat, vacuum distillation, behavior towards chemical agents, etc.[11]

Anthracite coal represents the final stage in the transformation of vegetable matter into a non-crystalline form. It contains definite proportions of hydrogen and oxygen. Certain forms of anthracite approach graphite in their composition. Graphite on the other hand is a crystallized mineral composed entirely of carbon, and which is supposed to represent the final stage in the metamorphosis of coal. The mode of occurrence and microscopic structure of graphite deposits corresponds closely with those of coal, giving rise to the belief that both were derived from a common source.

CHAPTER V

ANNUAL PRODUCTION OF BITUMINOUS SUBSTANCES AND THEIR MANUFACTURED PRODUCTS

PRODUCTION OF ASPHALTS, ASPHALTITES AND ASPHALTIC PYROBITUMENS

World Production. Deposits of natural asphalts have been discovered in all parts of the world. Table VIII, compiled from data furnished by the Department of Interior of the U. S. Geological Survey, shows the total production of all forms of native asphalts (including pure and rock asphalts), asphaltites and asphaltic pyrobitumens, from 1906 to 1931 inclusive, as far as reliable statistics are available.[1]

In 1931, the United States produced the largest quantity of native asphalts, having assumed the lead since 1920. Italy ranks second, followed by Trinidad, Germany, France and Venezuela, in the sequence stated.

Production in the United States. Table IX gives the total production by states in the United States of natural asphalts, asphaltites and asphaltic pyrobitumens for the years 1911 to 1935 inclusive, and Table X gives the production by varieties of natural asphalts, asphaltites, asphaltic pyrobitumens and petroleum asphalts from 1900 to 1935 inclusive.

No appreciable tonnage of asphalt was produced in the United States until 1883, when about 35,000 tons was imported mainly from Trinidad, which found its way into pavements. Prior to this, relatively small quantities of Trinidad was used, whereas European rock asphalts were imported for this purpose. In 1892 Trinidad asphalt had displaced most of the European products, and in that same year Bermudez asphalt made its appearance and gradually increased until 1919, when its tonnage approximated that of Trinidad asphalt.

The petroleum first known in the United States was derived from the Pennsylvania, Ohio and Indiana fields. This was of the paraffin type, and when distilled it left a viscous residue, more or less asphaltic in character, which was used principally to flux the harder native asphalts. This flux could not be distilled to a solid without decomposition and the formation of coke. In 1894 Byerley was granted a patent for converting these fluxes into semi-solid residues by blowing air through them at high temperatures, thus permitting them to be used without admixture for most purposes, although they never met with much favor in the paving industry. Upon the discovery of petroleum in California it was possible to obtain semi-solid to solid residual asphalts, closely resembling the native asphalts, and suitable for use in the construction of pavements. In 1902, petroleum asphalts found their way on the market in appreciable quantities, about 20,000 tons having been produced in that year. By 1911, the tonnage of domestic petroleum asphalts exceeded the importations of Trinidad and Bermudez asphalts combined, and from that time on the production of petroleum asphalts rapidly increased. In 1913 large quantities of Mexican petroleum asphalt found their way into the United States and rapidly increased until in 1918 it exceeded the domestic production. Since 1922, asphalt produced from Venezuela petroleum has been produced in rapidly increasing quantities, followed in 1925 by asphalt derived from Colombian petroleum.

At the present time petroleum asphalt far outranks other asphalts in importance. In 1935 the total tonnage of petroleum asphalt was almost ten times that of native asphalts and related substances. Of the total production, approximately 60 per cent was reported as having been derived from domestic crude petroleum, and 40 per cent from foreign crudes, including Venezuelan, Mexican and Colombian petroleum.

Table XI shows the production of petroleum asphalts by varieties in the United States in 1935, divided into two classes—solid and semi-solid products of less than 200 penetration and semi-solid and liquid products of more than 200 penetration. The major portion of the latter class is known by the general term "flux," which as the name implies, is used mainly as a blending material for the harder grades and also for blowing purposes.

TABLE VIII

Total World Production of Natural Asphalts, Asphaltites and Asphaltic Pyrobitumens (1906–1931)

(In Metric Tons)

	1906	1907	1908	1909	1910	1911	1912	1913	1914	1915	1916	1917	1918
Austria-Hungary	9,646	10,283	11,103	10,142	8,228	7,541	10,377	6,051	5,931	2,197	151(*a*)	259	(*b*)
Barbadoes	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Canada (Alberta)	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Cuba	5,186	5,054	6,237	10,795	2,105	3,300	15,658	1,587	879	441	489	473	450
France	196,321	177,026	171,111	169,008	169,722	169,651	311,763	41,471	35,555	11,707	14,381	12,068	10,104
Germany	117,380	126,614	88,985	77,516	81,186	81,880	96,117	105,460	81,756	32,434	31,541	12,321	7,980
Greece	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Italy	131,363	161,596	134,657	111,911	162,625	188,629	181,947	171,097	119,853	47,650	16,829	8,645	22,309
Japan	39	585	2,404	4,186	477	1,260	2,902	2,260	2,007	1,975	2,302	3,873	2,997
Netherland East Indies	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Peru	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12,080	8,131	14,915
Poland	(*c*)	(*c*)	(*c*)	(*c*)	(*c*)	(*b*)	(*b*)	1,353(*f*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)
Rumania	.....	.....	.....	.....	.....	.....	.....	.....	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)
Russia	11,355	12,806	22,644	2,418	24,988	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)
Spain	7,790	8,216	12,370	5,282	6,416	3,741	5,387	5,582	5,765	4,521	7,316	1,817	3,692
Switzerland	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	50,368	32,848	17,222	20,506	14,764	1,788
Trinidad	136,417	155,375	130,230	144,621	142,538	182,604	192,539	230,861(*d*)	112,800(*d*)	6,110(*e*)	130,847	134,034	72,550
United States	66,280	77,938	71,273	89,866	89,714	78,992	86,333	84,008	72,473	68,720	89,336	74,030	54,462
Venezuela	22,483	38,240	32,045	37,890	32,402	50,968	66,932	85,170	45,305	28,983	44,611	49,360	46,453
Yugoslavia	(*c*)	(*c*)	(*c*)	(*c*)	(*c*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)	(*b*)

	1919	1920	1921	1922	1923	1924	1925	1926	1927	1928	1929	1930	1931
Albania	.....	.....	.....	.....	.....	.....	.....	2,906	3,283	2,277	2,512	(b)	(b)
Austria	1,003	1,095	1,160	.....	.....	.....	.....	.....	.....	.....	.....	.....	(b)
Barbadoes	.....	92	111	133	152	71	30	109	46	60	51	10	9
Canada (Alberta)	.....	.....	.....	.....	.....	482	1,041	479	2,455	85	897	1,875	921
Cuba	(b)	3,043	342	109	2,098	3,643	1,534	8,682	4,116	13,186	10,695	16,064	(b)
Czechoslovakia	.....	.....	.....	.....	.....	.....	.....	.....	.....	586	1,783	669	1,011
France	18,117	25,024	39,595	48,477	40,292	51,491	52,467	58,267	61,014	65,308	63,693	56,548	57,734
Germany	12,554	31,904	29,352	54,619	46,244	57,359	64,115	60,966	117,836	158,329	145,341	117,161	(b)
Greece	.....	1,730	486	410	1,300	5,013	5,256	402	290	3,173	1,196	1,548	(b)
Irak	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	5,100	12,200	12,200
Italy	78,000	107,626	94,120	68,647	151,908	187,244	273,668	313,525	356,945	244,478	220,501	224,325	190,533
Japan	6,674	6,930	6,934	280	.....	.....	.....	.....	.....	.....	.....	.....	.....
Netherland East Indies	.....	431	365	(b)	(b)	650	670	155	1,340	1,050	15,030	8,778	2,394
Peru	14,033	17,162	11,834	8,829	(b)	2,708	1,068	690	892	981	950	(b)	(b)
Poland	299(f)	368(f)	260(f)	414(f)	710(f)	725(f)	738(f)	723(f)	736(f)	767(f)	835(f)	902(f)	261(f)
Rumania	750	1,056	14,122	13,016	21,967	19,731	15,712	24,891	30,712	8,875	.....	.....	.....
Russia	(b)	(b)	(b)	(b)	(b)	92(f)	11,680	32,589	49,221	49,330	(b)	(b)	(b)
Spain	4,564	4,222	3,533	5,933	3,936	5,476	5,580	5,856	4,514	7,735	9,002	9,480	5,135
Switzerland	8,888	16,653	17,394	22,045	19,910	33,159	35,757	28,200	36,716	21,321	20,009	17,119	15,331
Trinidad	95,523	143,501	121,651	185,587	224,900	227,460	204,335	241,109	246,017	199,125	223,128	160,393	125,114
United States	80,087	180,074	268,899	297,366	363,086	510,168	530,564	648,797	761,160	732,875	729,398	637,546	456,659
Venezuela	45,936	23,452	48,489	38,476	33,321	69,892	33,679	51,552	56,906	48,749	27,915	(b)	28,985
Yugoslavia	(b)	(b)	(b)	(b)	(b)	97	286	85	142	535	61	246	(b)

(a) Austria only. (b) Figures not available. (c) Included in Russia. (d) Fiscal year April 1st to March 31st. (e) Nine months, April 1st to December 31st.
(f) Principally Ozokerite.

TABLE IX

TOTAL PRODUCTION BY STATES IN THE UNITED STATES OF NATURAL ASPHALTS, ASPHALTITES AND ASPHALTIC PYROBITUMENS (1911–1935)

(In Short Tons)

	Utah (a)	Kentucky	Texas	Oklahoma	New Mexico	California	Kansas	Missouri	Alabama	Total
1911	30,846	6,000 (b)		13,747 (b)	.....	36,481	.....	.....	.....	87,074
1912	32,514	10,145		15,766	.....	36,741	.....	.....	.....	95,166
1913	30,810	17,465		16,459	.....	27,870	.....	.....	.....	92,604
1914	23,098	18,935		9,669	.....	28,186	.....	.....	.....	79,888
1915	21,739 (e)	19,311		16,907	.....	17,794	.....	.....	.....	75,751
1916	26,874	38,037		15,431	.....	18,135	.....	.....	.....	98,477
1917	35,192 (f)	34,610		5,793	.....	6,009	.....	.....	.....	81,604
1918	31,072 (g)	20,702 (b)		5,000 (b)	.....	3,260	.....	.....	.....	60,034
1919	33,992	46,352		4,323	.....	3,614	.....	.....	.....	88,281
1920	63,522	127,453 (c)		7,522	.....	.....	.....	.....	.....	198,497
1921	10,371	260,468 (c)		25,573	.....	.....	.....	.....	.....	296,412
1922	29,704	95,924 (b)	152,567	47,597	.....	2,000 (b)	.....	.....	.....	327,792
1923	34,635 (h)	184,300	151,356	25,800	.....	2,945	.....	.....	1,200	400,236
1924	36,536 (i)	274,743	220,598	18,261 (d)	.....	3,731	.....	.....	8,498	562,367
1925	39,790	286,850	204,530	27,450	.....	2,680	.....	.....	23,550	584,850
1926	42,430	320,430	289,980	37,010	.....	3,330	.....	.....	22,000	715,180
1927	42,940 (j)	344,220	376,770	39,460 (b)	.....	3,000 (b)	.....	.....	32,650	839,040
1928	57,447	318,548	321,505	110,360 (l)						807,860
1929	65,541	340,346	320,931	77,209 (l)						804,027
1930	47,635	305,024	270,138	79,980 (l)						702,777
1931	32,892	161,202	228,956	80,333 (m)						503,383
1932	25,980	91,289	132,636	90,114 (n)						340,019
1933	28,065	44,369	126,069	114,632 (n)						313,135
1934	30,399	90,027 (k)		290,940 (o)		29,486 (p)			.....	440,852
1935	33,227	109,179 (k)		185,013 (o)		19,917 (p)				347,336

(a) Includes the production of gilsonite, grahamite, wurtzilite and ozokerite.
(b) Figures estimated.
(c) Including California.
(d) Including 300 tons produced in Missouri.
(e) Including Colorado.
(f) Including 18 tons ozokerite.
(g) Including 37 tons ozokerite.
(h) Including 10 tons ozokerite.
(i) Including 300 tons ozokerite.
(j) Including 80 tons ozokerite.
(k) Including Alabama.
(l) Including Oklahoma, California and Alabama.
(m) Including Oklahoma, New Mexico, California and Alabama
(n) Including Oklahoma, New Mexico, California, Kansas, Missouri and Alabama.
(o) Including Texas, Oklahoma and New Mexico.
(p) Including California, Kansas and Missouri.

TABLE X

PRODUCTION BY VARIETIES IN THE UNITED STATES OF NATURAL ASPHALTS, ASPHALTITES, ASPHALTIC PYROBITUMENS AND PETROLEUM ASPHALT (1900–1935)

(In Short Tons of 2000 lb.)

Year	Natural Asphalts	Asphaltites		Asphaltic Pyrobitumens	Total Natural Asphalts, Asphaltites and Asphaltic Pyrobitumens	Petroleum Asphalts		
		Gilsonite	Grahamite	Wurtzilite		From Domestic Petroleum	From Foreign Petroleum	Total Domestic and Foreign
1900	.....	.....	.....	.....	54,389	.....	.....	.....
1901	.....	.....	.....	.....	63,134	.....	.....	.....
1902	.....	.....	.....	.....	84,632	20,826	.....	20,826
1903	.....	.....	.....	.....	55,068	46,187	.....	46,187
1904	59,639	2,978	1,000	550	64,167	44,405	.....	44,405
1905	49,982	10,916	1,500	500	62,898	52,369	.....	52,369
1906	58,163	12,447	1,952	500	73,062	64,997	.....	64,997
1907	64,240	20,285	966	422	85,913	137,948	.....	137,948
1908	57,296	18,533	2,286	450	78,565	119,817	.....	119,817
1909	66,278	28,669	3,894	220	99,061	129,594	.....	129,594
1910	70,061	25,432(*a*)	4,000 (*a*)	400 (*a*)	98,893	161,187	.....	161,187
1911	51,228	30,236	5,000	610	87,074	277,192	.....	277,192
1912	55,236	31,478	7,700 (*a*)	750 (*a*)	95,166	354,344	.....	354,344
1913	57,549	28,000(*a*)	6,500 (*a*)	700 (*a*)	92,604	436,586	114,437	551,023
1914	51,071	18,548(*a*)	9,669	600 (*a*)	79,888	360,683	313,787	674,470
1915	44,329	19,909(*a*)	10,863	650 (*a*)	75,751	664,503	388,318	1,052,821
1916	63,172	24,514(*a*)	8,431	2,360 (*a*)	98,477	688,334	572,387	1,260,721
1917	41,919	33,549(*a*)	4,618	1,518 (*ac*)	81,604	701,809	645,613	1,347,422
1918	25,346	30,848	3,803 (*b*)	37 (*d*)	60,034	604,723	597,697	1,202,420
1919	53,589	29,192(*a*)	5,000 (*a*)	500 (*a*)	88,281	614,692	674,876	1,289,568
1920	132,353	56,204	9,940 (*b*)	.....	198,497	700,496	1,045,779	1,746,275
1921	284,037	10,066	2,004	305	296,412	624,220	908,093	1,532,313
1922	298,047	29,693	41	11	327,792	805,145	1,242,163	2,047,308
1923	365,601	34,425	.....	210 (*e*)	400,236	995,654	1,378,722	2,374,376
1924	525,831	35,907	60	569 (*f*)	562,367	1,158,456	1,920,915	3,079,371
1925	545,060	39,520	.....	270	584,850	1,206,700	1,971,670	3,178,370
1926	672,750	42,190	.....	240	715,180	1,245,160	2,213,310	3,458,470
1927	796,100	42,580	.....	360 (*g*)	839,040	1,525,420	2,426,030	3,951,450
1928	760,497	47,023	.....	340 (*h*)	807,860	1,930,536	2,298,848	4,229,384
1929	748,550	54,987	.....	490 (*i*)	804,027	2,332,973	2,355,498	4,688,471
1930	664,871	37,684	.....	222	702,777	1,403,552	1,824,089	3,227,641
1931	470,491	32,763	.....	129	503,383	1,274,744	1,700,946	2,975,690
1932	314,039	25,955	.....	25	340,019	1,115,547	1,359,372	2,474,919
1933	285,070	28,029	.....	36	313,135	1,237,386	1,218,665	2,456,051
1934	410,453	30,355	.....	44	440,808	1,444,846	1,395,650	2,840,496
1935	314,109	33,227	.....	61	347,336	1,801,778	1,485,225	3,287,003

(*a*) Estimated.
(*b*) Including wurtzilite.
(*c*) Including 18 tons ozokerite.
(*d*) Including 37 tons ozokerite.
(*e*) Including 10 tons ozokerite.
(*f*) Including 300 tons ozokerite.
(*g*) Including 80 tons ozokerite.
(*h*) Including 150 tons ozokerite.
(*i*) Including 290 tons ozokerite.

TABLE XI

ASPHALT AND ASPHALTIC MATERIAL (EXCLUSIVE OF ROAD OIL) SOLD AT PETROLEUM REFINERIES IN THE UNITED STATES, IN 1935, BY VARIETIES

[Value f. o. b. refinery]

	From Domestic Petroleum		From Foreign Petroleum		Total	
	Short Tons	Value	Short Tons	Value	Short Tons	Value
Solid and semisolid products of less than 200 penetration: *						
Asphalt for:						
Paving	419,771	$3,994,713	457,695	$4,805,877	877,466	$8,800,590
Roofing	303,234	3,170,647	328,138	3,585,501	631,372	6,756,148
Waterproofing	83,294	917,623	65,821	703,711	149,115	1,621,334
Blending with rubber	3,187	40,663	11,944	136,081	15,131	176,744
Briquetting	37,674	416,870	3,271	39,305	40,945	456,175
Mastic and mastic cake	192	1,718	2,310	25,393	2,502	27,111
Pipe coatings	6,212	113,311	1,728	16,963	7,940	130,274
Molding compounds	6,856	75,914	7,814	85,016	14,670	160,930
Miscellaneous uses	47,749	500,746	56,836	639,325	104,585	1,140,071
Total	908,169	$9,232,205	935,557	$10,037,172	1,843,726	$19,269,377
Semisolid and liquid products of more than 200 penetration: *						
Flux for—						
Paving	67,393	$581,032	45,673	$432,539	113,066	$1,013,571
Roofing	225,195	1,540,995	54,649	616,485	279,844	2,157,480
Waterproofing	3,736	58,480	24,074	299,767	27,810	358,247
Mastic	.....	.....	32	320	32	320
Cut-back asphalts	378,054	3,927,834	329,899	3,481,445	707,953	7,409,279
Emulsified asphalts and fluxes	22,412	239,789	17,449	119,861	39,861	359,650
Paints, enamels, japans, and lacquers	12,216	162,620	8,528	83,321	20,744	245,941
Other liquid products	91,942	398,208	17,624	142,605	109,566	540,813
Total	800,948	$6,908,958	497,928	$5,176,343	1,298,876	$12,085,301
Grand total, 1935	1,709,117	$16,141,163	1,433,485	$15,213,515	3,142,602	$31,354,678
Total, 1934	1,353,639	$13,973,765	1,373,602	$15,921,674	2,727,241	$29,895,439

* DEFINITIONS

Paving asphalt.—Refined asphalt and asphaltic cement, fluxed and unfluxed, produced for direct use in the construction of sheet asphalt, asphaltic concrete, asphalt macadam, and asphalt block pavements, and also for use as joint filler, in brick, block, and monolithic pavements.

Roofing asphalt.—Asphalt and asphaltic cement used in saturating, coating, and cementing felt or other fabric in the manufacture of asphalt shingles.

Waterproofing asphalt.—Asphalt and asphaltic cement used to waterproof and dampproof tunnels, foundations of buildings, retaining walls, bridges, culverts, etc., and for constructing built-up roofs.

Briquetting asphalt.—Asphalt and asphaltic cement used to bind coal dust or coke breeze into briquets.

Mastic and mastic cake.—Asphalt and asphaltic cement for laying foot pavements and floors, waterproofing bridges, lining reservoirs and tanks, capable of being poured and smoothed by hand troweling.

Pipe coatings.—Asphalt and asphaltic cement used to protect metal pipes from corrosion.

Molding compounds.—Asphalts used in the preparation of molded composition, such as battery boxes, electrical fittings, push buttons, knobs, handles, etc.

Miscellaneous uses.—Asphalt and asphaltic cement used as dips, and in the manufacture of acid-resisting compounds, putty, saturated building paper, fiber board and floor coverings, and not included in the preceding definitions.

Flux.—Liquid asphaltic material used in softening native asphalt or solid petroleum asphalt for paving, roofing, waterproofing, and other purposes.

Cut-back asphalts.—Asphalts softened or liquefied by mixing them with petroleum distillates.

Emulsified asphalt and fluxes.—Asphalts and fluxes emulsified with water for cold-patching, road laying, and other purposes.

Other liquid products.—Petroleum asphalt, exclusive of fuel oil used for heating purposes, not included in the preceding definitions.

ROAD OIL SOLD BY PETROLEUM REFINERIES IN THE UNITED STATES, 1934–35, BY DISTRICTS

District	1934		1935	
	Barrels	Value	Barrels	Value
East coast	938,053	$1,392,665	1,001,845	$1,614,179
Appalachian	88,195	186,298	34,035	64,437
Indiana–Illinois–Kentucky	1,984,414	2,390,175	1,957,569	2,439,100
Oklahoma–Kansas–Missouri	942,072	1,071,260	597,450	547,789
Texas:				
Gulf coast	204,888	$274,188	99,969	$153,069
Rest of State	79,969	79,963	22,772	35,775
Total Texas	284,857	$354,151	122,741	$188,844
Louisiana–Arkansas:				
Louisiana Gulf coast	52,464	$ 95,089	68,203	$103,334
Northern Louisiana and Arkansas	157,992	158,104	384,769	357,745
Total Louisiana and Arkansas	210,456	$253,193	452,972	$461,079
Rocky Mountain	1,023,434	$1,431,920	644,485	$1,032,907
California	2,231,272	3,662,336	1,987,835	2,194,621
Grand total	7,702,753	$10,741,998	6,798,932	$8,542,956

PRODUCTION OF TARS AND PITCHES

Tars Derived from Coal. By far the most important source of tar produced in the United States is derived from bituminous coal. The following represents the production in United States gallons:[2]

TABLE XII

	Gas Works (Horizontal, Vertical and Inclined)	Coke Ovens (All Types)	Water and Oil Gas
1904	42 million	28 million	9 million
1908	58	43	9
1912	41	94	34
1916	50	180	55
1918	53	263	101
1919	53	289	105
1920	51	361	116
1921	50	253	109
1922	48	328	104
1923	48	441	...
1924	48	422	108
1925	49	480	...
1926	50	529	105
1927	51	547	...
1928	52	632	110
1929	47	681	117
1930		602	
1931		451	
1932	50 to 40 (est'd)	304	110 to 80 (est'd)
1933		363	
1934		409	
1935	40 (est'd)	468 (est'd)	85 (est'd)

No statistics are available giving the production of the corresponding types of pitch.

Tars and Pitches Derived from Wood. The following figures have been reported by the U. S. Department of Commerce:[3]

	Wood Tar (gallons)	Wood-tar Pitch (tons)
1923	5,500,000	.....
1925	9,000,000	13,500
1927	8,500,000	10,000
1929	10,500,000	4,500

Other Tars and Pitches. There are no published figures giving the annual production of other tars and pitches in the United States. The following estimates have been compiled by the author as a mere approximation of what he believes to be their present relative standing.

	Tons Annually
Rosin pitch	20,000 to 30,000
Fatty-acid pitch	15,000 to 20,000
Blast-furnace tar and pitch	5,000 to 10,000
Bone tar and bone-tar pitch	2,000 to 5,000
Shale and lignite tars and pitches	250 to 500

MANUFACTURED PRODUCTS

Bituminous Paving Materials. The total tonnage of all types of asphalt, including asphalt cements, fluxes, cut-back asphalts, emulsified asphalts and liquid residual asphaltic products used for highway purposes in the United States have been reported as follows:[4]

Year	Tons
1925	1,764,700
1926	1,800,180
1927	2,179,100
1928	2,487,642
1929	2,655,989
1930	2,693,552
1931	2,901,851
1932	2,838,344
1933	2,500,110
1934	2,977,990
1935	3,225,000

The mileage of improved roads completed and under construction as of January 1st of the current year has been estimated to be as follows:[5]

	1935 Miles	1936 Miles	1937 Miles
Sand-clay treated	4,079	7,502	5,605
Gravel treated	22,893	27,725	43,194
Macadam treated	31,664	24,635	17,756
Low-cost bituminous mixture	33,120	39,872	45,555
Bituminous macadam	16,593	16,466	16,555
Bituminous concrete	10,717	11,572	11,583

Bituminous Roofing Products. Table XIII compiled from statistics furnished by the Asphalt Shingle and Roofing Institute, New York City, and the U. S. Department of Commerce, Washington, D. C., show the sales of asphalt roll-roofings and asphalt shingles in the United States from 1917 to 1936.

TABLE XIII

SHIPMENTS IN "SALES" SQUARES—TOTAL INDUSTRY U. S. A.

Year	Smooth-roll Roofings	Slate-roll Roofings	Strip Shingles	Individual Shingles	Total All Types
1917	21,252,000	4,134,000	1,266,600	1,625,000	28,277,000
1918	19,250,000	4,312,000	1,017,000	1,149,000	25,728,000
1919	16,930,000	6,428,000	2,311,600	2,183,000	27,952,600
1920	17,579,000	6,624,000	2,253,900	1,866,000	28,322,000
1921	13,992,000	7,670,000	2,865,230	1,632,000	26,159,230
1922	14,604,000	9,252,000	4,243,630	2,395,000	30,494,630
1923	12,558,000	9,847,000	5,602,150	2,492,000	30,499,150
1924	11,951,450	10,608,100	6,707,950	3,103,000	32,570,500
1925	11,990,709	10,529,521	7,803,202	3,406,975	33,730,411
1926	13,502,977	11,061,229	8,529,382	3,209,728	36,303,316
1927	14,783,909	10,798,454	9,810,918	2,582,031	37,975,312
1928	16,074,654	9,608,380	9,002,297	1,854,393	36,539,724
1929	17,896,549	9,989,787	9,959,382	2,015,781	39,861,499
1930	12,359,414	7,147,670	6,820,775	1,546,238	27,874,097
1931	10,794,478	5,563,338	4,887,134	1,319,752	22,564,702
1932	11,996,723	5,493,513	4,143,133	1,130,522	22,763,891
1933	13,837,632	5,684,905	4,223,069	991,547	24,737,153
1934	12,837,702	5,489,467	4,422,486	1,255,346	24,005,001
1935	12,485,950	6,426,369	5,350,006	1,787,826	26,050,151
1936	15,251,646	8,005,678	6,851,860	2,118,724	32,227,908

The statistics in Table XIV have been compiled by the U. S. Department of Commerce, Washington, D. C.:[6]

TABLE XIV

PRODUCTS, BY KIND, QUANTITY AND VALUE, 1929, 1931 AND 1935

Kind	1935	1931	1929
Roofing, built-up and roll; asphalt shingles; roof coatings other than paint, all products	$76,172,740	$58,962,919	$103,506,090
Roofing	70,589,616	49,453,352	94,591,578
Other products (not normally belonging to the industry)	5,517,081	9,509,567	8,914,512
Receipts for contract and custom work	66,043	.....	.....
Roofing, built-up and roll, asphalt shingles, and roof coatings other than paint, made as secondary products in other industries	3,434,376	6,143,744	6,493,283
Asphalt roll roofing:			
Total roofing squares	22,331,481	17,525,143	28,437,076
Total value	$24,768,468	$20,272,517	$36,109,653
Smooth-surfaced:			
Roofing squares	14,373,813	11,659,193	18,718,704
Value	$13,710,544	$11,213,050	$20,032,452
Grit-surfaced:			
Roofing squares	7,957,668	5,865,950	9,718,372
Value	$11,057,924	$9,059,467	$16,077,201
Asphalt shingles:			
Total roofing squares	7,731,034	6,558,524	11,676,636
Total value	$26,801,952	$22,566,749	$42,291,343
Strip:			
Roofing squares	6,048,383	5,206,259	9,692,774
Value	$21,897,294	$18,328,528	$33,707,384
Individual:			
Roofing squares	1,682,651	1,352,265	1,983,862
Value	$4,904,658	$4,238,221	$8,583,959
Saturated felt:			
Total tons	298,219	143,512	205,525
Total value	$8,042,840	$6,025,401	$9,803,629
Asphalt-saturated felt:			
Tons	271,241	110,727	137,026
Value	$6,237,267	$4,685,835	$6,631,461
Tar-saturated felt:			
Tons	26,978	32,785	68,499
Value	$1,805,573	$1,339,566	$3,172,168
Waterproofing fabrics, value	$409,417	(a)	(a)
Roof-coatings and cements, total value	$6,374,359	$4,298,946	$6,311,449
Asphalt roof cement (solid):			
Tons	106,790	16,630	35,161
Value	$1,420,651	$333,750	$842,420
Coal-tar roofing pitch:			
Tons	47,509	(a)	(a)
Value	$898,898		
Fibrous plastic roof-cement:			
Pounds	31,469,942	29,488,861	52,753,395
Value	$1,176,998	$1,347,599	$2,336,111
Fibrous liquid roof-coating:			
Gallons	4,881,023	4,979,013	4,941,563
Value	$1,928,595	$2,040,783	$2,115,578
Nonfibrous liquid roof-coating:			
Gallons	3,424,671	1,420,622	1,931,269
Value	$949,217	$576,814	$1,017,340
Other roofing materials, value	$6,383,417	$2,433,483	$6,568,787
Asphalt brick siding:			
Squares	277,730	(a)	(a)
Value	$1,243,539		

(a) No data.

TABLE XIV—*Continued*

PRODUCTS, BY KIND, QUANTITY AND VALUE, 1929, 1931, 1933 AND 1935

Kind	Year	Quantity	Value
		Squares	
Asbestos shingles (*a*)	1935	1,171,095	$5,234,727
	1933	408,256	1,826,279
	1931	565,997	3,266,054
	1929	894,164	5,277,308
Asbestos flexible roofing (*a*), (*b*)	1935	890,854	567,911
Asphalt roll roofing (*c*)	1935	22,331,481	24,768,468
	1933	12,333,342	12,763,519
	1931	17,525,143	20,272,517
	1929	28,437,076	36,109,653
Asphalt shingles (*d*)	1935	7,731,034	26,801,952
	1933	4,008,811	11,994,618
	1931	6,558,524	22,566,749
	1929	11,676,636	42,291,343
		Tons (2000 lbs.)	
Asphalt and tar saturated felt (*d*)	1935	298,219	$8,042,840
	1933	75,623	2,778,308
	1931	143,512	6,025,401
	1929	205,525	9,803,629
		Squares	
Clay roofing tile (*e*)	1935	253,951	1,145,434
	1933	103,257	810,647
	1931	285,253	3,125,175
	1929	370,771	3,943,847
		Tons (2000 lbs.)	
Concrete roofing tile (*f*)	1935	(*g*)	(*g*)
	1933	11,971	338,809
	1931	31,860	862,197
	1929	137,052	2,869,092
Steel sheets, corrugated and crimped (*h*):		*Tons (2240 lbs.)*	
Galvanized	1935	358,574	27,611,766
	1933	243,542	15,922,000
	1931	262,307	18,381,000
	1929	306,423	26,363,000
Not galvanized	1935	6,506	542,981
	1933	2,338	140,000
	1931	3,026	175,000
	1929	5,285	380,000
		Thousands	
Wood shingles (*i*)	1935	3,526,318	10,358,360
	1933	2,929,800	6,958,275
	1931	2,713,972	4,993,708
	1929	6,110,672	18,026,482
		Tons (2000 lbs.)	
Asphalt roof-cement (solid) (*c*)	1935	106,790	1,420,651
	1933	10,373	255,471
	1931	16,630	333,750
	1929	35,161	842,420
		Pounds	
Fibrous plastic roof-cement (*c*)	1935	31,469,942	1,176,998
	1933	13,968,939	661,044
	1931	29,488,861	1,347,599
	1929	52,753,395	2,336,111
		Gallons	
Fibrous liquid roof-coating (*c*)	1935	4,881,023	1,928,595
	1933	3,003,734	1,011,323
	1931	4,979,013	2,040,783
	1929	4,941,563	2,115,578
Nonfibrous liquid roof-coating (*c*)	1935	3,424,671	949,217
	1933	799,844	183,729
	1931	1,420,622	576,814
	1929	1,931,269	1,017,340

(*a*) Made in the Asbestos Products industry. (*b*) No figures available for earlier years. (*c*) Made principally in the Roofing Materials industry. (*d*) Made in the Roofing Materials industry. (*e*) Made in the Clay Products industry. (*f*) Made in the Concrete Products industry. (*g*) Not yet available. (*h*) Made principally in the Steel Works and Rolling Mills industry. (*h*) Made in the Lumber and Timber Products industry.

Asphalted Felt-base Floor Coverings. The data in Table XV have been compiled by the U. S. Department of Commerce, Washington, D. C.:[7]

TABLE XV

ASPHALTED FELT-BASE FLOOR COVERING—PRODUCTS, BY KIND, QUANTITY AND VALUE

	1935	1931	1929
Total square yards	128,043,256	87,575,642	117,969,725
Total value	$31,259,943	$21,628,899	$36,943,057
Piece goods:			
Total square yards	66,630,594	48,401,157	57,026,360
Total value	$14,461,266	$11,341,150	$14,680,017
12/4 and wider:			
Total square yards	20,704,429	5,210,590	6,960,553
Total value	$4,412,620	$1,227,276	$2,032,865
Made on less than .050-inch-gauge felt:			
Square yards	12,330,005	2,444,984	2,169,111
Value	$2,448,789	$502,930	$509,821
Made on .050-inch-gauge felt or thicker:			
Square yards	8,374,424	2,765,606	4,791,442
Value	$1,963,831	$724,346	$1,523,044
8/4:			
Total square yards	43,766,063	38,886,096	44,400,278
Total value	$9,531,524	$9,110,304	$11,621,078
Made on less than .050-inch-gauge felt:			
Square yards	24,644,425	22,824,979	23,809,293
Value	$5,124,065	$4,584,784	$5,470,632
Made on .050-inch-gauge felt or thicker:			
Square yards	19,121,638	16,061,117	20,590,985
Value	$4,407,459	$4,525,520	$6,150,446
Narrower than 8/4:			
Total square yards	2,160,102	4,304,471	5,665,529
Total value	$517,122	$1,003,570	$1,026,074
Made on less than .050-inch-gauge felt:			
Square yards	1,224,073	3,658,997	5,494,659
Value	$285,894	$807,106	$965,874
Made on .050-inch-gauge felt or thicker:			
Square yards	936,029	645,474	170,870
Value	$231,228	$196,464	$60,200
Rugs:			
Total square yards	61,412,662	39,174,485	60,943,365
Total value	$16,798,677	$10,287,749	$22,263,040
Made on less than .050-inch-gauge felt:			
Square yards	33,741,412	25,348,060	27,299,303
Value	$8,342,774	$5,760,542	$7,758,888
Made on .050-inch-gauge felt or thicker:			
Square yards	27,671,250	13,826,425	33,644,062
Value	$8,455,903	$4,527,207	$14,504,152

PART II

SEMI-SOLID AND SOLID BITUMENS AND PYROBITUMENS

CHAPTER VI

METHODS OF MINING, TRANSPORTING AND REFINING

MINING METHODS

Three general methods are followed in mining semi-solid and solid bituminous substances, depending upon the nature of the deposit.

Open-cut Quarrying. Deposits at or near the surface are mined by simple quarrying methods. If exposed on hillsides, the rock asphalt is blasted loose and loaded on railroad cars by means of steam shovels or buckets running on a cable way. Deposits located beneath flat terrain are mined by open cuts, after first removing the overburden, as illustrated in Figs. 46 and 49. The asphalt may be hauled from the cut by means of buckets suspended from an overhead cable way, or by dump-cars pulled up inclined tracks on cables.

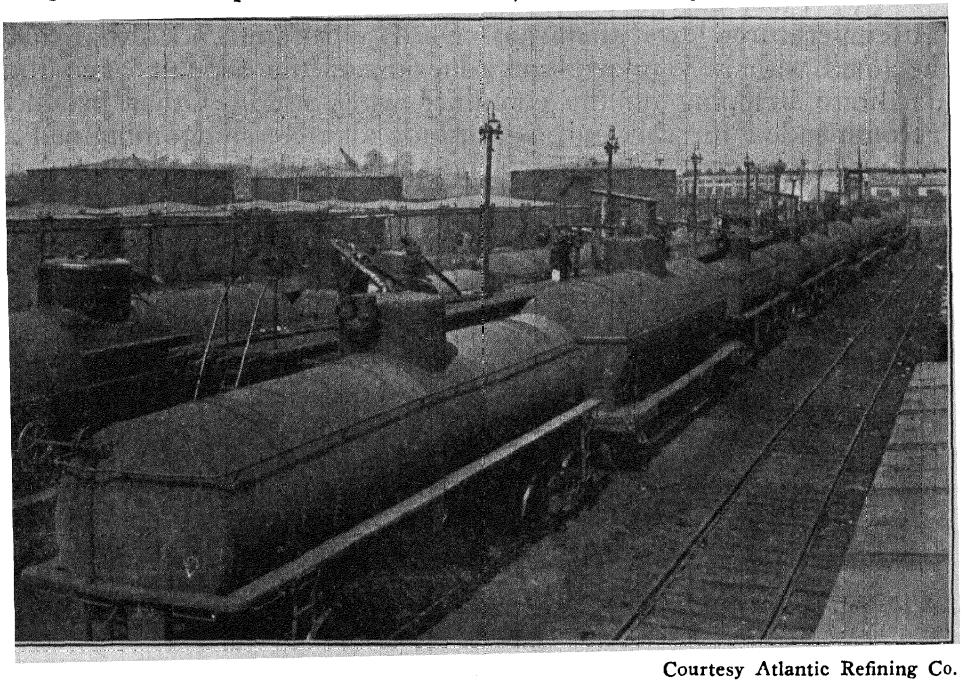
Tunnelling. Veins situated a distance beneath the surface are mined by tunnelling methods, which also adapt themselves to handling veins filling hillside faults or fissures. Typical tunnelling methods are illustrated in Figs. 64 and 69. Horizontal veins may be handled by pit-and-stall methods if the overlying stratum is solid enough to resist caving in, otherwise the roof and possibly also the sides of the tunnel must be timbered, which of course adds to the cost of mining. Asphaltites are generally mined in this manner.

Special Methods. Asphalts deposits occurring in the form of so-called lakes, as for example at Trinidad and Bermudez, are mined by comparatively crude methods, involving the use of the pick and shovel, as illustrated in Figs. 43 and 59. The asphalt is dumped

by hand in small cars on a movable track, whence it is transported to the main railroad line or to steamers, if near the seacoast.

METHODS OF SHIPMENT AND TRANSPORTATION

Hard rock asphalts and asphaltites are shipped in fragments, either in bulk the same as coal, or if the material is of sufficient intrinsic value to warrant the expense, it may be shipped in sacks weighing 150 to 200 lb. each. Where intended for constructing asphalt mastic pavements or floors, the rock asphalt is cast in the



Courtesy Atlantic Refining Co.

FIG. 35.—Tank-cars Used for Transporting Asphalt.

form of flat cakes weighing between 50 and 75 lb. for the convenience of handling.

If the asphalt is comparatively free from mineral matter, and this applies also to petroleum asphalt, it may be shipped in six types of containers, as follows:[1]

(1) In tank cars having capacities of 6500, 8000 and 10,000 gal. as illustrated in Fig. 35. These are provided with steam coils for remelting the asphalt upon arrival at destination, and are preferably insulated, so that they may be transported in a melted state with the minimum loss of heat.

(2) Tank trucks equipped with heating flues in which a fire is maintained by an oil burner during transit. These are generally used for transporting asphalts for road and paving purposes.

(3) Light metal drums with a metal top having a 6-in. hole in the center for convenience in filling. These usually weigh 475 to 525 lb., holding 50 to 55 gal.

(4) Wooden barrels of about the same capacity as stated in (3). These are generally used for transporting liquid to semi-liquid materials, and are filled at the lowest temperature to prevent excessive contraction taking place upon cooling.

(5) Fiber barrels and other containers,[2] which should be filled at temperatures lower than 300° F. It is recommended that these be lined with clay to prevent adhesion of the asphalt[3] and thus facilitate stripping off the container when used. Other products recommended for this purpose include alkaline sludge obtained as a by-product in refining petroleum products,[4] glycerol foots,[5] an aqueous solution of oxalic acid or sodium-hydrogen phosphate containing glycerol,[6] etc.

(6) Molds for the shipment of high melting-point asphalts, which may be of the same form as barrels. These are filled in the same manner as drums, and when the asphalt has cooled, they are opened up and the block removed and shipped as such.[7] The cakes may also be coated with a high fusing-point asphalt or asphaltite (e.g., gilsonite).[8] This method eliminates the payment of freight on the container, but only adapts itself to materials which are sufficiently rigid and tough to withstand shipment.

METHODS OF REFINING

Dehydration. Most native asphalts contain more or less moisture, which may be present either accidentally as hydroscopic moisture, or in the form of an emulsion. Trinidad asphalt is an example of the latter, in which about 29 per cent of water is emulsified with the asphalt and clay.

Before the asphalt can be used commercially, this water or moisture must be removed. The process by which this is accomplished is known as "dehydration." The expulsion of water is brought about by heating the asphalt in a suitable open container constructed of iron or steel, which is built in two types, viz.:

(1) Semi-cylindrical.
(2) Rectangular.

In either case the top is left open so that the water may be expelled readily. In modern plants, the heating tanks are built to contain between 10 and 30 tons of the crude asphalt.

The heating is effected by various means:

(1) By direct fire heat, in the form of a combustion chamber underneath the tank, enclosed in fire bricks. Three kinds of fuel are used for this purpose, depending upon which is most readily obtained in the locality where the asphalt is to be refined; namely, coal or coke, oil, or gas. Coal is burnt on a grate; oil is usually

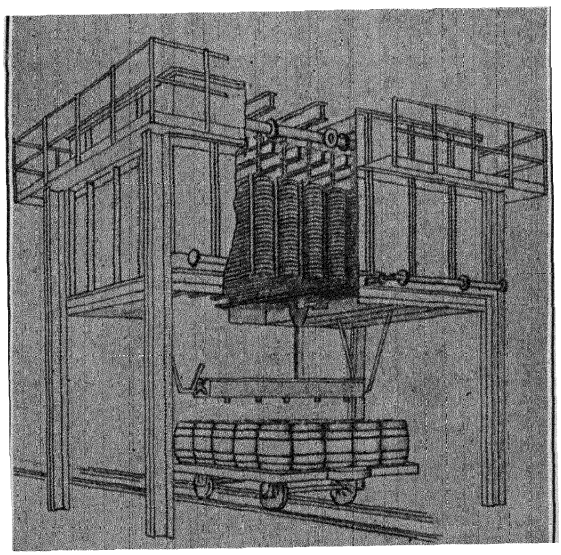
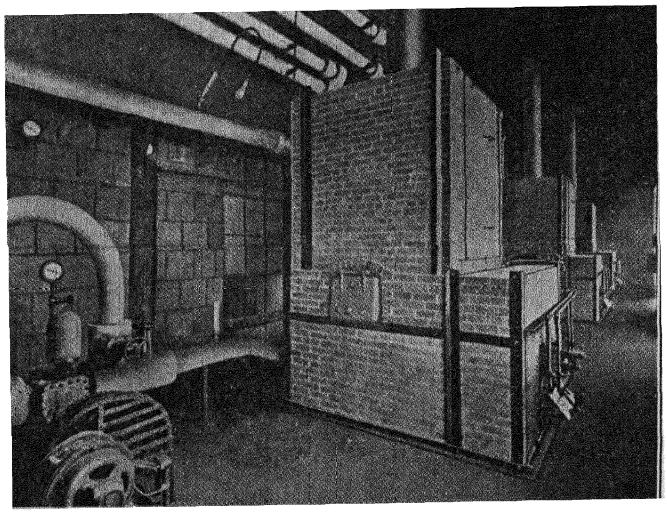


FIG. 36.—Showing Arrangement of Refining Stills for Natural Asphalt.

sprayed into the combustion chamber by compressed air or steam; and natural or producer gas is introduced through a suitable type of burner.[9] In any case, the best practice consists in protecting the bottom of the melting-tank by a fire-brick arch work, so that the hot gases are compelled to circulate back and forth.[10] This subjects the bottom of the tank to a more uniform temperature, and tends to prolong its life. At the same time, it economizes fuel by more thoroughly extracting the heat from the hot gases, due to the increased area of contact with the bottom of the tank. Some recommend the use of a perforated brick arch to distribute the hot gases uniformly and prevent the bottom of the tank from being overheated locally. Fire melting-tanks are usually semi-cylindrical in form, although sometimes they may be rectangular at the top, with a semi-cylindrical bottom.

(2) By means of steam. In this case the heating is effected by coils of steam pipes contained in the tank. One and one-quarter to 1½-in. pipes are generally used for this purpose. According to the best practice, these are bent in coils composed of a continuous length of pipe without unions or joints, as illustrated in Fig. 36.[11] Another method consists in using cast-iron headers with straight lengths of 1¼ or 1½-in. pipes fastened in between. Steam is used at pressures between 125 and 150 lb. This will raise the temperature of the asphalt to 300 or 400° F. Steam has the advantage over fire heat in not coking the asphalt, which would tend to insulate the bottom, induce local overheating, and burn out the tank in a comparatively short time.



Courtesy of Parks Cramer Co.

FIG. 37.—Hot Oil Circulation Method for Heating Asphalts.

(3) Electrical Immersion Heaters.[12] Electric heating units contained in a series of 10-in. pipes (closed at both ends) are placed at the bottom of the tank. Each unit has a capacity of 54 k.w. and serves to heat the air in the pipe, which in turn imparts its heat to the surrounding asphalt. This same device has been utilized for saturating roofing felt, in which case the asphalt is maintained at 440° F. while the felt is being treated at the rate of 1 ton per hour, which corresponds to a heat consumption of about 10,000 B.t.u. per hour per ton of asphalt.

(4) By contact with molten metals or alloys. Another procedure consists in promoting the rapid heating of the asphalt by a bath of molten lead, tin, zinc, or various alloys of low fusing-point.[13]

(5) By hot oil circulation. This is accomplished by circulating heated mineral oil of a high flash-point through a closed system of pipes, including a heating coil in the asphalt melting tank and a second coil located in a furnace situated a short distance away from the tank. The second coil is heated by means of an oil or gas burner, which by means of a thermostatic control serves to heat the oil to a predetermined temperature, usually at 550 to 600° F. Fig. 37 illustrates the oil heater and circulating pump.[14]

(6) By means of diphenyl vapor. Diphenyl is a white solid, melting at 156.6° F. and boiling at 491.5° F. under a pressure of 1 atmosphere. At 491.5° F. it has a pressure of 14.7 lb. absolute and at 600° F. it has a pressure of only 47 lb. In view of these properties, its use has been promoted for heating asphalts, etc.[15] A mixture of diphenyl and diphenyl oxide has also been proposed,[16] likewise naphthalene.

(7) By circulating the asphalt itself.[17] This is a modification of the preceding method, in which the mineral oil is replaced by the asphalt which is to be heated. In this case the heating coil in the asphalt melting tank is omitted, and the asphalt is pumped directly from the bottom of the tank through a heating coil in the outside furnace, and then back again into the melting tank. The heating coil is brought to the proper temperature (which may be controlled thermostatically) by oil or gas combustion. This procedure is simple and efficient, but adapts itself only to asphalts having a high flash-point and which are substantially free from mineral matter or other ingredients which will settle in the coils and induce carbonization. The asphalt must also be free from water and capable of being heated rapidly without frothing.

The time of heating can be reduced materially by agitating the asphalt mechanically, since the transfer of heat through a mass of asphalt is very slow. The agitation may be accomplished:

(*a*) By jets of dry steam which should be introduced after the temperature of the asphalt becomes sufficiently high to prevent condensation, and thus avoid excessive foaming.[18]

(*b*) By jets of air.

(*c*) By mechanical agitators.

During the process of dehydration, the mass is apt to froth when the temperature is raised beyond the boiling-point of water. For this reason, it is well to build the tanks large enough to accommodate the foam without danger of overflowing. Shallow tanks are preferable to deep tanks.

Certain types of asphalt are most difficult to dehydrate, as they foam very badly. Numerous devices have been used to keep down the foam, the simplest and most successful consisting in directing a current of hot air against the surface of the asphalt while it is being melted.

The use of steam accelerates the evaporation of the more volatile constituents in the asphalt, and is therefore apt to cause a greater shrinkage during the dehydration than when air or mechanical mixing is used.[19] On the other hand, air is apt to "oxidize" the asphalt and increase its fusing-point, especially if its use is continued for long periods of time.

Sometimes the asphalt is subjected to a process of partial distillation in a closed retort to remove the volatile constituents and raise its fusing-point, which, however, is stopped before the formation of carbonaceous matter.[20] A modification consists in adding a proportion of vegetable oil during the refining process for the purpose of absorbing the sulfur derivatives loosely combined with the asphalt.[21]

Any impurities such as vegetable matter, chips of wood, etc., which rise to the surface when the asphalt is melted should be skimmed off. When the asphalt is thoroughly melted and the foaming ceases, the dehydration is complete. It is usually unnecessary to raise the temperature of the asphalt higher than 350° F. The dehydrated asphalt may be discharged:

(1) By a valve at the bottom of the tank, permitting the asphalt to flow out by gravity.

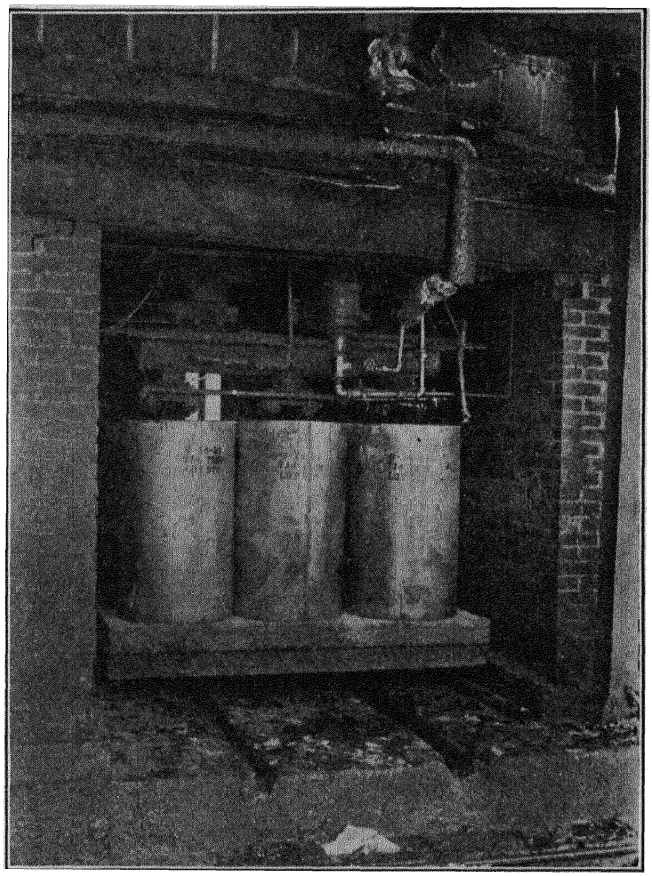
(2) By a rotary pump which may either be steam-jacketed[22] or surrounded by a steam coil in close contact with the pump, the entire installation being well insulated. The rotary pump is usually installed above the level of the asphalt, and the intake pipe extended almost to the bottom of the heating-tank.

(3) By means of a pneumatic lift installed below the bottom of the tank. The asphalt is allowed to flow by gravity into the pneumatic lift, which, by a suitable mechanism, automatically shuts off the flow when it is filled, and then admits compressed air, forcing the asphalt upward through the discharge pipe. The pneumatic lift may either be steam-jacketed or heated with a steam coil as described.

(4) By means of an Archimede's screw, which serves to extrude asphalt compositions in the plastic state.[23]

A device for discharging the melted asphalt into light metal drums, in which nine are filled simultaneously, is illustrated in Fig. 38.

Asphalt may be pumped through pipe lines for distances of 500 feet or more. To effect this it must be maintained in a melted state. This is accomplished by running a steam pipe of small



Courtesy Atlantic Refining Co.

FIG. 38.—Discharging Melted Asphalt into Drums.

diameter inside the pipe carrying the asphalt.[24] The outer pipe should be well insulated.

Hard asphalts are first pulverized and then dried in air.[25]

Distillation. Attempts have also been made to utilize rock asphalts associated with a small percentage of bituminous matter, and which cannot be utilized for other purposes, by subjecting them

to a process of destructive distillation [26] and recovering the volatile products, including the lubricating oil fraction,[27] which correspond closely with those obtained upon distilling pyrobituminous shales.

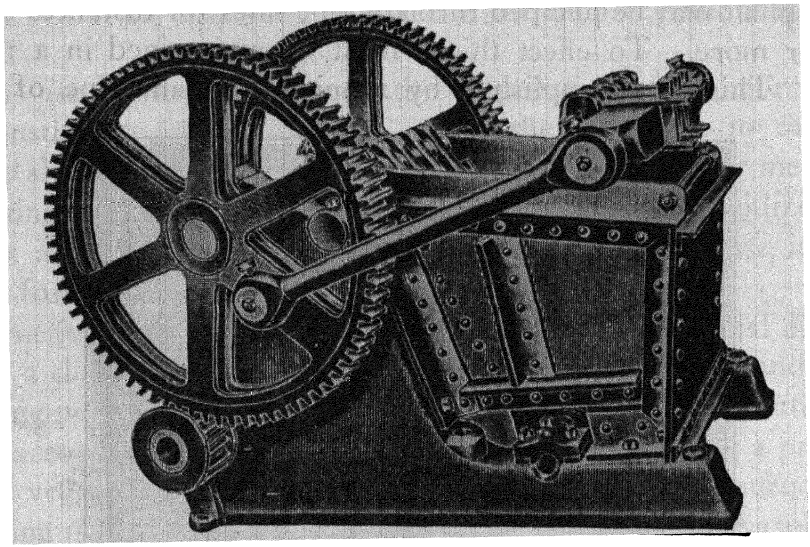


FIG. 39.—Jaw Crusher.

However, the low market price of petroleum has been an obstacle to the success of such operations.

Rock asphalts may be distilled to harden same for use in paving

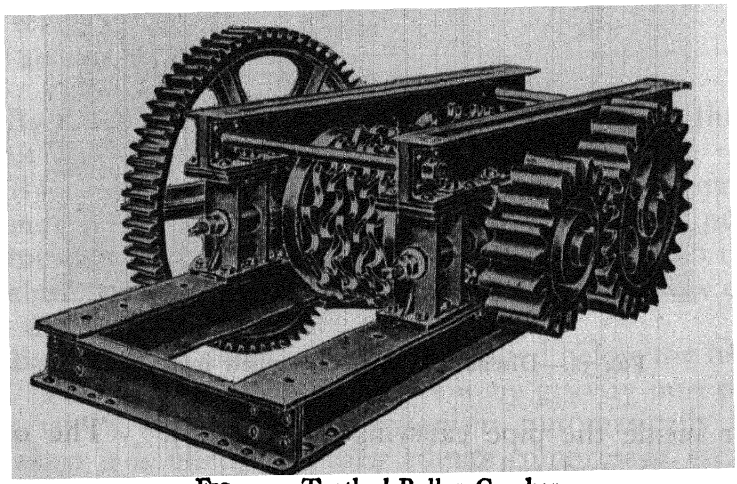


FIG. 40.—Toothed-Roller Crusher.

compositions.[28] If coarse or undesirable mineral aggregate is present, it may be screened to the desired mesh while in a melted state, and electrically heated screens have been proposed for this purpose.[29]

Comminution. Natural rock asphalts and asphaltites may be comminuted [30] in three different types of apparatus, including jaw-crushers illustrated in Fig. 39, toothed rollers as illustrated in Fig.

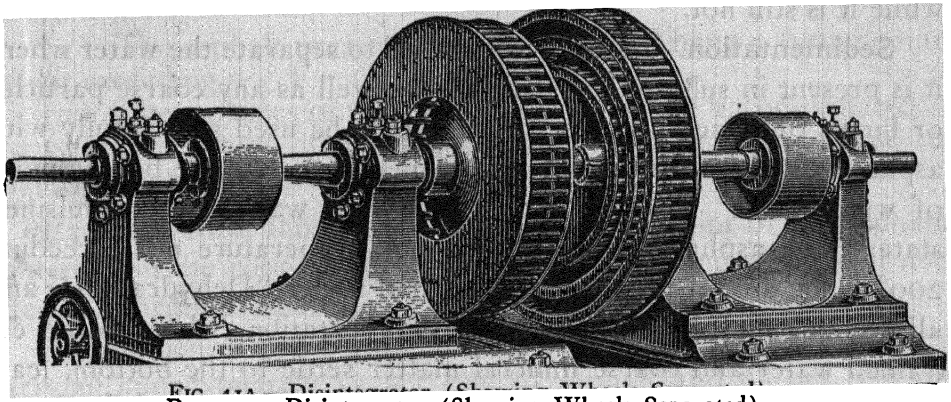


FIG. 41A.—Disintegrator (Showing Wheels Separated).

40, and a disintegrator as illustrated in Figs. 41 (A) (showing the machine taken apart) and 41 (B) (showing the machine assembled). After being crushed and ground, the product is screened to the re-

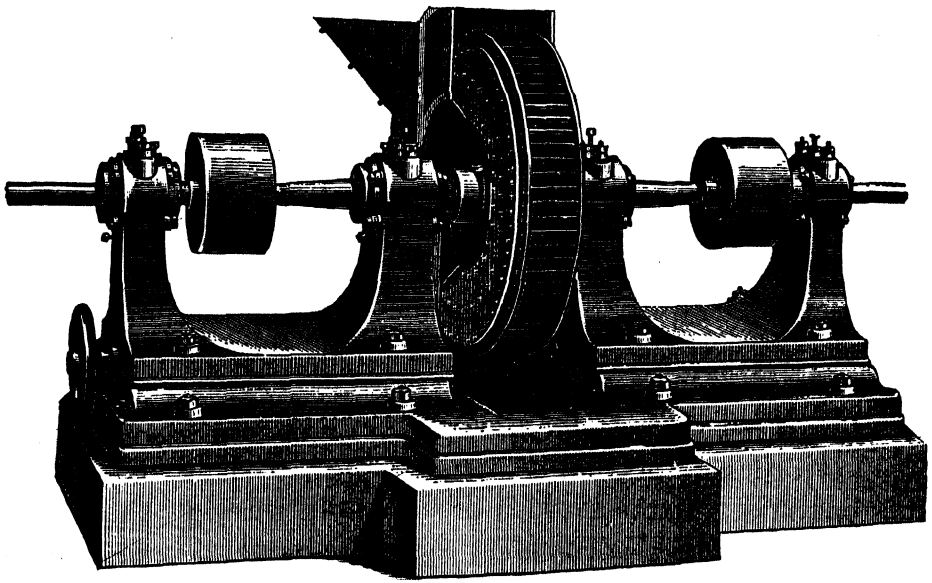


FIG. 41B.—Disintegrator (Showing Machine Assembled).

quired mesh, and finally heated in a revolving, fire-heated cylinder to expel the associated moisture, usually at a temperature of 260 to 300° F. The asphalt content of the comminuted material can be

adjusted by grinding together rock taken from the rich veins with material mined from the poorer strata. When used for paving purposes, the product is usually transported directly to the job while it is still hot.[31]

Sedimentation. This process is used to separate the water where it is present in substantial quantities, as well as any coarse particles or lumps of mineral matter. It can only be used successfully with asphalts or other forms of bitumen melting below the boiling-point of water (212° F.), and not carrying the water in an emulsified state. The asphalt is maintained at a temperature not exceeding 200° F. by any of the devices described under "Dehydration," and allowed to undergo a process of sedimentation, whereby the entrained water and coarse mineral matter settle to the bottom, leaving the purified asphalt on top. The latter is then carefully drawn off.[32] Steam heating is most satisfactory for this purpose.

In some cases only a portion of the water separates by sedimentation, whereupon the process is supplemented by one of dehydration. The sedimentation will remove most of the water and has the advantage of materially shortening the dehydration process. A combination of the two processes will thus prove more effective than the use of either one alone.

Since water usually has a higher specific gravity than melted asphalt, it tends to settle to the bottom of the vessel containing it. This invariably proves to be the case with the softer forms of native asphalt.

Extraction. Two media have been used for this purpose, namely water and volatile solvents. As the methods are entirely different, they will be considered separately.

Extraction by Means of Water. This method has been used with more or less success for extracting asphalt from asphaltic sands, sandstone and limestone. It is based on the principle that water has a higher specific gravity than the melted asphalt, and a lower gravity than the accompanying mineral matter, so that when boiled together, the melted asphalt will rise to the surface and the mineral constituents settle to the bottom.[33] Calcium chloride,[34] sodium carbonate [35] and salt [36] have been proposed to be added to the water to increase its gravity and thereby effect a more thorough separa-

tion of the asphalt. Rock asphalts after weathering yield more difficultly to the water-separation process.[37]

To yield successfully to this method, the rock asphalt must possess the following characteristics:

(1) The asphalt present in the rock should have a fusing-point of not exceeding 90° F. (Test 15*a*.)
(2) The particles of mineral matter should be unconsolidated.
(3) The grains of mineral matter should be fairly coarse to enable them to settle rapidly.

Experience has shown that when the fusing-point of the asphalt contained in the rock is higher than 90° F., boiling water will not effect a thorough separation.

A specimen of asphaltic sand obtained near Woodford, Okla., carrying approximately 12 per cent of asphalt and 88 per cent of sand in the form of loose, rounded grains between 40- and 80-mesh, separated fairly completely on boiling with water. The pure asphalt showed a fusing-point between 65 and 70° F.

Another asphaltic sand obtained near Fort McMurray, in northern Alberta, carrying approximately 15 per cent of asphalt and 85 per cent of non-compact sand, between 40- and 100-mesh, likewise largely separated on boiling with water. The fusing-point of the pure asphalt was 50° F.

The same was true with an asphaltic sand carrying 17 per cent of asphalt obtained from a deposit 46 miles northwest of Edmonton, and 12 miles north of Onoway. In this case the pure asphalt fused at 62° F.

A Mexican asphaltic sand carrying 16 per cent of asphalt also separated completely on boiling with water, the fusing-point of the pure asphalt being 78° F.

On the other hand, certain asphaltic sands obtained from various localities of Oklahoma, carrying between 10 and 15 per cent of asphalt, refused to separate on boiling with water. The fusing-points of the pure asphalts were found to be 113° F., 118° F., and 127° F., respectively. The particles of the sand were substantially similar to the preceding, ranging between 40- and 100-mesh.

Plants for the water-extraction of asphalt from rock asphalt have been in operation in Oklahoma (sand asphalt); Texas (asphaltic limestone); Alberta, Canada (sand asphalt); Pechelbronn,

Alsace-Lorraine (asphaltic limestone); Seyssel and Bastennes, France (asphaltic limestone); San Valentino, Italy (asphaltic limestone); Tataros, Austria (asphaltic limestone); also in Russia (sand asphalt).

A cross-sectional diagram of an apparatus which gives fairly successful results is shown in Fig. 42. The separated asphalt must be treated in accordance with the methods described under the heading "Dehydration," to separate the water which is mechanically carried along with it. If the process has been performed properly, the purified asphalt will not contain more than 5 to 7 per cent of mineral matter. The water-extraction process also is used for puri-

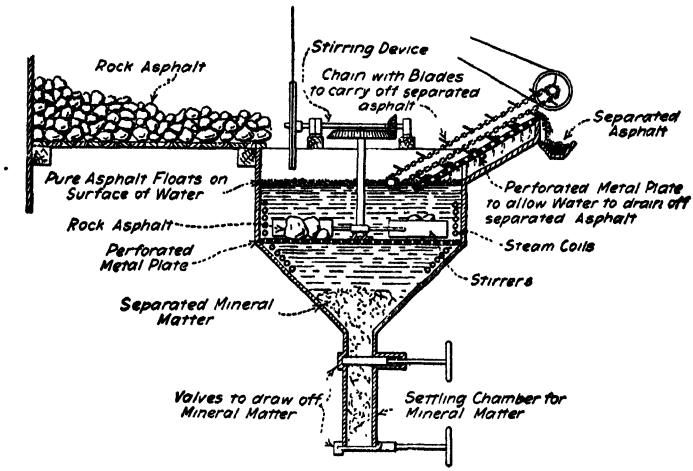


FIG. 42.—Apparatus for Separating Soft Asphalt from Sand by Means of Water.

fying ozokerite and to remove water-soluble constituents (e.g., salt) from certain native asphalts, and thereby improve their weather-resisting properties.[38]

A flotation process has been proposed, which consists in agitating the sand asphalt at 60 to 80° C. with a 0.1 per cent solution of a froth former, e.g., soda ash, alkaline soap, turkey-red oil, saponin, glue, etc.; the aqueous solution being then separated by decantation and the mineral matter removed by lixiviation.[39] The use of bentonite [40] alkali (e.g., NaOH or Na_2CO_3),[41] sodium phosphate,[42] sodium silicate,[43] sodium silicate in combination with calcium chloride or salt,[44] sulfonated mineral oils,[45] etc., have also been suggested for this purpose.

Extraction or Precipitation with Solvents. Carbon disulfide, petroleum distillates and benzol have been used for this purpose. This method has not proven successful commercially, on account of its expense. Several plants have been constructed in the United States for extracting asphalts from asphaltic sands and sandstone. The Alcatraz Asphalt Co. of Alcatraz, Cal. (1896–1899), erected an elaborate plant for treating rock carrying 10 to 16 per cent of asphalt. The venture, however, proved a failure through losses in solvent (a light distillate of petroleum), which made the cost of treatment prohibitive. The loss was due in part to unavoidable evaporation during the extraction process, also to the impossibility of fully recovering the solvent from the extracted residue.

Solvents may be used either to extract solid bituminous substances (e.g., rock asphalts, sand asphalts, certain pitches, etc.), or to precipitate components from fluid bituminous substances (e.g., petroleum, tars, etc.) or solutions of solid bituminous substances in various menstra. The following classes of solvents have been proposed for these purposes:[46]

(a) Solvents derived from petroleum:

Crude petroleum;[47] liquid ethane, pentane, propane or butane;[48] light petroleum distillates (e.g., petroleum ether, gasoline, or petroleum naphtha);[49] kerosene;[50] heavy mineral oils;[51] etc.

(b) Solvents derived from coal tar:

Benzol;[52] toluol;[53] xylol (solvent naphtha);[54] heavy oils;[55] naphthalene;[56] tetrahydronaphthalene ("tetralin");[57] nitrobenzene;[58] benzonitrile;[59] phenol;[60] phenyl nitrile;[61] aniline;[62] pyridine or quinoline.

(c) Solvents derived from wood:

Turpentine;[63] methyl or ethyl alcohol;[64] higher alcohols (e.g., butyl, isobutyl, propyl, iso-propyl, amyl, fusel oils, benzyl, etc.);[65] acetone;[66] acetone oils;[67] wood oils;[68] furfural or methyl furfural ("furfural process");[69] glacial acetic acid;[70] methyl or ethyl acetate;[71] amyl acetate; croton aldehyde ("Foster-Wheeler process").

(d) Sulfur derivatives:

Liquid sulfur dioxide ("Edeleanu process");[72] carbon disulfide;[73] sulfo-derivatives of mineral oils and tars.[74]

(e) Sundry solvents:

Ethyl ether;[75] carbon tetrachloride, trichlorethylene, ethylene dichloride, or dichlor-diethyl ether ("chlorex process"); amine bases (e.g., iso-amyl amine, di-iso-propyl amine, di-iso-butyl amine, etc.).[76]

(f) Mixtures of solvents:

Petroleum naphtha with amyl alcohol;[77] pentane with butane, or propane with ethane;[78] propane with cresol ("Dusol process"); benzol or toluol with alcohols;[79] benzol with acetone;[80] benzol with liquid sulfur dioxide ("Edeleanu process"); propane with liquid sulfur dioxide;[81] ethyl alcohol with ethyl ether; methyl formate with benzol, carbon disulfide or carbon tetrochloride;[82] phenol with a polyhydric alcohol;[83] successively with alcohol, or acetone, petroleum naphtha, and finally with benzol or toluol.[84]

For the production of high-grade lubricating oils from heavy petroleum fractions, the use of liquid propane as a solvent (or more accurately, as "antisolvent") has become increasingly popular.[85] The following constituents present in ordinary lubricating oil fractions must be eliminated, viz.: paraffin wax to obtain a low pour-point, asphalt because of its instability and excessive carbon-forming tendencies, the heavy ends of the lubricating oils because of their high carbon-forming tendencies, naphthenic compounds of olefinic or aromatic characteristics because of their low stability and low viscosity index, and color bodies to render the lubricating oil more marketable. Refining with liquid propane makes available as by-products a whole series of high melting-point waxes, also petrolatums of extremely high quality, and new types of asphalt of excellent ductility and penetration relations. The same is true to a certain extent with other light hydrocarbons, including liquid ethane, which dissolves much less oil than liquid propane and throws asphalt completely out of solution. A mixture of methane and propane behaves similarly to ethane. Liquid butane, on the other hand is too good a solvent to be effective in removing the asphalt. For the complete removal of wax, asphalt, heavy ends, naphthenic compounds and color bodies, liquid propane is generally more efficient than any other solvent known at present.

Propane gas, by simple compression and liquefaction, may be converted into a solvent having the unique properties (under proper conditions of temperature and pressure) of separating each of the foregoing undesirable constituents. It owes its versatility to the fact that its properties change rapidly over the particular temperature range between $-44°$ and $+215°$ F. Over this range, it possesses the properties of a series of solvents, any one of which can be obtained by raising or lowering the temperature, or chang-

ing the pressure, or by combining these two operations. Pure propane boils at — 44° F. under 1 atmosphere pressure, exerts a pressure of 126 lb. per sq. in. at 70° F., and has a critical pressure of 643 lb. at 212.2° F. Over this range propane changes from a typical liquid to a fluid possessing substantially the properties of a gas. Between 70° and — 44° F., its main uses are in connection with dewaxing. At temperatures above 70° F., it finds its use as a preferential precipitant. Thus, in the range of 100° to 140° F., asphalt is only slightly soluble in propane, whereas at these same temperatures both oil and wax dissolve completely. Between 100° and 212° F. and at the vapor pressure of propane, instead of behaving as an ordinary liquid and dissolving more of any partially soluble substances as the temperature is raised, it actually dissolves less. The heavier and more naphthenic compounds are thrown out first, but as the properties of propane become increasingly those of gases, it dissolves less and less oil, until no viscous oil remains in solution at 212.2° F.—the critical temperature of propane. This property makes it possible to recover by simple decantation a large proportion of the propane in a substantially pure state, without the necessity to vaporize it by the usual method.

To utilize these varying properties of propane on a stock containing all the foregoing undesirable constituents, a typical cycle consists in mixing the oil with 4 to 6 times its volume of liquid propane at 120° to 150° F., which will throw out of solution practically all of the asphalt. The latter may be settled out and drawn off as a liquid because it contains a substantial quantity of dissolved propane. The remaining solution can then be rapidly chilled by the simple expedient of lowering the pressure and allowing a small quantity of the propane to evaporate until a temperature of — 20° to — 40° F. is attained, whereupon the wax will be thrown out of solution and may be removed by settling or preferably by filtration. The propane solution is then brought back to room temperature, and may either be treated with relatively small quantities of sulfuric acid to remove the color bodies and naphthenic constituents, or else be heated without any additions, in stepwise fashion, to about 200° F. Under these conditions a series of cuts of varying composition is separated out; the first is composed of very heavy ends, resinous constituents, and the more heavy naph-

thenic constituents of the oil, and each cut becomes lighter and more paraffinic, until the last material left in solution is a relatively low-boiling, highly paraffinic, light-colored oil.

The term "propane de-asphalting" is usually applied to those cases where the heavy product is a mixture containing black, pitchy material. This operation may include removal of only a small amount (approximately 3.5 per cent) of high melting-point asphalt from a Mid-continental crude, to a much larger percentage from a California crude. In the latter case, the separated asphalt had a specific gravity at 60° F. of 1.031, a penetration at 77° F. of 35, a fusing-point (R. and B. method) of 118° F., and over 99.6 per cent soluble in CS_2, CCl_4 and 86° petroleum naphtha, respectively.

Attempts have also been made, but without success, to utilize the solvent extraction method to recover the bituminous constituents and felt respectively from the waste material produced in the manufacture of asphalt roofings and shingles.[86] On the other hand, it has given satisfactory results in recovering montan wax from lignite at Thuringia, Saxony, and from pyropissite at Weissenfels, near Halle, Germany. Benzol is generally used for this purpose, although in certain cases petroleum distillates have given good results. The high market price of montan wax in comparison to asphalt undoubtedly accounts for this.

METHODS OF STORAGE

Bituminous substances which are capable of liquefying under heat are usually stored in metal tanks of various capacities. These may be equipped with steam-coils. One device consists in constructing a small metal tank within a larger one, the smaller unit being provided with the steam coils to conserve the heat.[87]

CHAPTER VII

MINERAL WAXES

OZOKERITE

Ozokerite is a native mineral wax, composed of the higher members of the C_nH_{2n+2}, and C_nH_{2n} series of hydrocarbons. It occurs in deposits usually associated with petroleum. Certain varieties carry a proportion of petroleum in solution with the wax, and the more petroleum present, the softer will be the consistency and lower the fusing-point. Ozokerite as ordinarily found is fairly hard, and has a comparatively high fusing-point, ranging from 50 to 180° F. The fusing-point has been recorded as high as 200° F. (K. and S. method). Ozokerite containing between 10 and 15 per cent of petroleum in solution, shows a fusing-point between 140 and 150° F. The petroleum can readily be evaporated by applying a moderate degree of heat, and is expelled during the refining process.

The color of ozokerite depends upon the nature and extent of the impurities present, and ranges from a transparent yellow to a dark brown. In rare instances, ozokerite occurs in a dichroic variety, showing a dark green color by reflected light, and a pure yellow by transmitted light. It breaks with a conchoidal fracture, and has a characteristic waxy lustre. Its streak on porcelain varies from a transparent white to a pale brown.

Ozokerite is usually found filling veins or fissures, which are very irregular in structure, varying from a fraction of an inch to about two feet in thickness. Some extend for comparatively long distances whereas others pinch out very suddenly. The veins are usually caused by faulting, which accounts for their irregularity and gives the vein the appearance of a series of pockets. The indications are that the ozokerite enters the faults or veins from below, which is borne out by the fact that the material mined at a depth is materially softer and has a lower fusing-point than that obtained near the surface.

Ozokerite may occur in a pure state (comparatively free from

mineral matter), which proves to be the case when it is found in vein form, or it may be associated with sandstone or shale. In fact, it is quite common for the entire region surrounding the vein to be saturated with ozokerite.

A paraffinaceous petroleum almost invariably occurs in the strata underlying the ozokerite, which would seem to indicate that the latter must have been produced by the slow hardening and probably also the oxidation of petroleum throughout centuries of time. Ozokerite, itself, however, is practically free from oxygen. In this particular case, therefore, the effect of oxidation is to eliminate hydrogen, and form hydrocarbons of higher molecular weight.

The petroleum underlying the ozokerite usually contains from 8 to 12 per cent of paraffin, which, however, is entirely different in its character from the hydrocarbons contained in the ozokerite.[1] This proves conclusively that ozokerite is not formed merely by the evaporation of petroleum, but must have been produced by a process of metamorphosis or polymerization.

On distilling at atmospheric pressure, ozokerite decomposes, whereas when it is distilled under reduced pressure, its composition changes but little. Still another process consists in distilling the ozokerite in retorts with superheated steam, whereupon the residue in the retort is known as "ozokerite pitch," which when combined with rubber has been marketed under the name "okonite."

Ozokerite as it is mined is assorted by hand-picking to separate the pure material from that associated with earthy matter. The latter, which is known as "wax-stone," is broken up to remove any lumps of rock and purified by extraction with boiling water. The method used for this purpose is very crude, and consists merely in boiling the wax-stone with water in large open kettles. The sandstone or shale separates to the bottom and the melted ozokerite floats in a layer on the surface, whereupon it is skimmed off, boiled to evaporate the water and cast into blocks. The commercial material is comparatively free from mineral matter, rarely containing over 2 per cent.

Ozokerite may be refined still further by heating to 120–200° C., with 20 per cent by weight of concentrated sulfuric acid, or with chromic acid,[2] or by treating ozokerite with alkali and filtering hot through fuller's earth, animal charcoal, or magnesium silicate. This

bleaches the ozokerite, forming a product almost white in color, known as "ceresine." From 10 to 15 per cent of the ozokerite is lost during the treatment, but the fusing-point of the product is increased. The final traces of acid are removed and the bleaching process completed by adding from 5 to 12 per cent of dry residue obtained from the manufacture of "ferrocyanides." The main differences between ozokerite and ceresine are in the color and fusing-point.

Ozokerite and ceresine are used in the manufacture of high-grade candles, colored lead pencils, for finishing off the heels and soles of shoes, manufacturing shoe polishes, electrical insulating purposes, as an acid-proof coating for electrotypers' plates, and waxing floors. They are readily soluble in turpentine, petroleum distillates, carbon disulfide, and benzol, by scarcely soluble in alcohol.

Purified ozokerite and ceresine comply with the following characteristics:

(Test 1)*	Color in mass	White to yellow to brown
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Dull to "waxy"
(Test 6)	Streak	Transparent white to yellow
(Test 7)	Specific gravity at 77° F.	0.85–1.00
(Test 9*a*)	Hardness, Moh's scale	Less than 1
(Test 9*b*)	Penetration at 32° F.	0
	Penetration at 77° F.	20–30
	Penetration at 115° F.	150–250
(Test 9*c*)	Consistometer hardness at 32° F.	Above 100
	Consistometer hardness at 77° F.	20–40
	Consistometer hardness at 115° F.	5–15
(Test 9*d*)	Susceptibility factor	Greater than 80
(Test 15*a*)	Fusing-point (K. and S. method)	140–200° F.
(Test 15*b*)	Fusing-point (R. and B. method)	155–225° F.
(Test 19)	Fixed carbon	½–10 per cent
(Test 21)	Soluble in carbon disulfide	95–100 per cent
	Non-mineral matter insoluble	0–1 per cent
	Mineral matter	0–5 per cent
(Test 22)	Carbenes	0–3 per cent
(Test 23)	Non-mineral matter soluble in 88° petroleum naphtha	75–95 per cent
(Test 26)	Carbon	84–86 per cent
(Test 27)	Hydrogen	16–14 per cent
(Test 28)	Sulfur	0–1.5 per cent
(Test 29)	Nitrogen	0.0.5 per cent
(Test 30)	Oxygen	0–2 per cent
(Test 33)	Solid paraffins	50–90 per cent
(Test 34*b*)	Sulfonation residue	90–100 per cent
(Test 37*e*)	Saponifiable constituents	0–2 per cent

* The numbers refer to tests, which are described in detail in Chapter XXXII.

Very often ozokerite and ceresine are adulterated with paraffin wax, rosin, tallow, stearic acid or mineral fillers (such as talc, kaolin, gypsum, etc.).

Ozokerite occurs in the following localities:[3]

EUROPE

Poland (Galicia).[4] The most important ozokerite deposits are found in the Carpathian Mountains in the districts of Drohobycz (comprising Boryslaw, Wolanka and Truskawiec) and Stanislau (comprising Dwiniacz, Straunia, Wolotkow and Niebylōw). It is known under various names, e.g., "ozokerit," "fossil or montan wax," "mineral fat," etc. In the Moldau region it is called "zietriszit," and around the Caspian Sea, "nephtgil," "neftgil," "naphatil," etc. The largest deposit is located as Boryslaw, a small town in Galicia, and has been exploited since about 1859. It is found at some depths below the surface, associated with schist and sandstone, and it is mined by means of shafts and galleries. About 1500 shafts have been sunk in the district.

The following varieties of ozokerite are recognized in the Boryslaw district:

(1) Marble wax, found 100–200 m. below the surface, is very hard, of a pale yellow color, with greenish, brownish and black markings, giving it the appearance of marble, and has a fusing-point of 85 to 100° C.

(2) Hard wax or "crackwax" (Sprungwax) is darker in color than marble wax, shows a granular fracture and a fusing-point of 75 to 90° C.

(3) Fibrous wax, or "fibrewax," which is characterized by its fibrous structure.

(4) Bagga is dark in color, contains clay, and has a comparatively low fusing-point (40–60° C.).

(5) Kindebal or "kinderball" is characterized by being soft, of low fusing-point (30–50° C.) and a black color. It contains petroleum and mineral matter.

(6) Blower wax ("blister wax" or "matka") is a pale yellow, soft variety, which is squeezed out of the veins due to the presence of the surrounding rocks.

(7) Lep is a variety of ozokerite associated with a substantial proportion of mineral matter.

The deposit at Wolanka is smaller than that at Boryslaw. The

occurrence at Truskawiec differs from the others by the presence of a comparatively large percentage of sulfur. The ozokerite in this locality is associated with native sulfur, lead sulfide, gypsum, and petroleum.

At Dwiniacz, Straunia and Wolotkow, about 70 miles south of Boryslaw, the ozokerite veins are located some distance below the surface, in beds of clay between layers of shale. Considerable ozokerite has been mined in this district, and particularly at Dwiniacz. The veins vary in size from ¼ in. to about 1 ft. The rock in the vicinity of the veins is impregnated with wax, containing an average of 2 per cent.

Rumania. Deposits of ozokerite are also found in a spur of the Carpathian Mountains in the province of Moldavia in Rumania. It has been mined in several localities, the largest vein occurring in the city of Slanik, beneath a bed of bituminous shale, associated with a vein of cannel coal. This deposit is characterized by its high fusing-point, in the neighborhood of 200° F. (K. and S. method).

Russia. Numerous deposits of ozokerite occur throughout Russia, including specifically the following:[5]

Terek Province (*Northern Caucasia*). In the vicinity of Groznyi, among the narrow passes along the road from Wozdwishensk and Chatoi, near the right bank of the Chanti-argun River.

Kuban Province (*Northern Caucasia*). In the oil fields at Maikop, near Chadychenskaja, on so-called Wax Mountain, about 5 km. from Pshish stream.

Kutais Province (*Transcaucasia*). In the vicinity of Kutais, 2 km. east of the village Dznucisi, between the rivers Lechidary and Merchery; likewise at Achokrua in the same neighborhood; also near Charopan, northeast of the village Tedeleti, in a gorge between the mountains Gagberula and Syrch-Liberta.

Tiflis Province (*Transcaucasia*). At Sadzluri, in the neighborhood of Cori, on the banks of Kran River.

Baku Province (*Transcaucasia*). In the Baku oil fields, near the town Surachany, also on the Island of Suyatoi in the Caspian Sea.

Kars Province (*Transcaucasia*). In the region of Chala-Darasi, in the vicinity of Kars. Occurs in layers containing 1.5 to 2.5 per cent ozokerite in Uzbekistan, at Sel-rokho and Shorsu.

ASIA

State of Turkestan. On the Island of Cheleken in the Caspian Sea;[6] also in the vicinity of Ferghana, also at Neftedag (Turkoman Republic) in veins containing 50 to 80 per cent ozokerite, termed locally "lep."

Siberia. Along the eastern shore of Lake Baikal.

Philippine Islands. On the Island of Leyete, veins of ozokerite occur at the contact of an intrusive dike with shales and sandstones.[7]

NORTH AMERICA

UNITED STATES

Utah. The most important deposit occurs near Colton, in Wasatch County, Utah,[8] in a bed of oil shale. The veins extend from about 2 miles west of Colton to within a few hundred yards west of the railroad station of Soldier Summit, or a total distance of 12 miles. The mineral as mined contains about 15 per cent ozokerite which is extracted by heating the crushed ore with water at 60–70° C., whereupon the wax floats off. A peculiar feature of this deposit is the occurrence of fossil shells together with other animal remains. The following analytical results have been reported:

(Test 1)	Color in mass	Yellowish brown
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Pale yellow
(Test 7)	Specific gravity at 77° F	0.899–0.920
(Test 9*a*)	Hardness, Moh's scale	Less than 1
(Test 9*b*)	Penetration at 77° F	30
	Penetration (100 g., 15 secs.):	
	at 20° C	0.43 mm.
	at 30° C	0.64 mm.
	at 40° C	1.27 mm.
	at 50° C	2.2 mm.
	at 60° C	3.8 mm.
(Test 12*i*)	Coefficient of expansion between 10 and 50° C., per deg. C	0.001
(Test 15*f*)	Fusing-point	60 to 80° C
(Test 16)	Volatile, 212° F. 1 hr	0.0 per cent
	Volatile, 325° F., 5 hrs	45.41 per cent
	Volatile, 400° F., 5 hrs	65.2 per cent
(Test 19)	Fixed carbon	9.6 per cent
(Test 21)	Solubility in carbon disulfide	99.46 per cent
	Non-mineral matter insoluble	0.50 per cent
	Free mineral matter	0.046 per cent

(Test 22)	Carbenes	2.51	per cent
(Test 23)	Soluble in 88° petroleum naphtha	81.71	per cent
(Test 26)	Carbon	85.35	per cent
(Test 27)	Hydrogen	13.86	per cent
(Test 28)	Sulfur	0.29	per cent
(Test 29)	Nitrogen	0.36	per cent
(Test 58)	Dielectric strength across a 0.1-in. gap	over 30,000	volts
	Dielectric constant	2.03	
	Power factor	0.03	
	Resistivity across a 1-cm. cube	30 million	megohms

Texas. At Thrall, in the so-called Thrall Oil Field, another deposit has been reported.[9] The crude material is soft, due to the petroleum associated with it, and of a dark brown color. It has a strong odor of petroleum, and a specific gravity at 77° F. of 0.875. On being heated to 100° C., it loses 14.72 per cent, and at 180° C. a total of 23.14 per cent in weight. On being freed from petroleum, it shows a fusing-point of 175° F.

Hatchettite or Hatchettine

The above names are assigned to a soft variety of ozokerite fusing in the neighborhood of 120° F. (Test 15*f*). It varies in specific gravity from 0.90 to 0.98 at 77° F., and has a yellowish-white, yellow or greenish-yellow color. It was named after C. Hatchett, an English chemist (1765–1847). It is found near Merthyr-Tydvil in Glamorganshire, England, also at Loch Fyne in Argylshire, Scotland.

Scheererite

A native wax found in a bed of lignite near St. Gall, Switzerland. It occurs in the form of crystals (monoclinic) and of a white, gray, yellow, green or pale reddish color. It is more or less translucent to transparent, and has a waxy feel. It is composed chiefly of the members of the paraffin series, and fuses at a temperature of 110–115° F.

Kabaite

This is a waxy hydrocarbon similar to ozokerite or scheererite, which has been found in meteorites. It is only of scientific interest and has no commercial importance.

Montan Wax

As stated previously, montan wax is dissolved from certain non-asphaltic pyrobitumens by means of volatile solvents, such as benzol, xylol, mixtures of benzol with methyl alcohol or acetone,[10] mixtures of toluol and alcohol,[11] etc. By extracting under pressure the yield is materially increased, and at 50 atmospheres at 250–260° C., benzol will extract about double the weight than at 1 atmosphere at 80° C.[12] The lignite or pyropissite is first dried, then granulated, and finally extracted. The extract is evaporated to recover most of the solvent. The last traces of solvent are expelled from the montan wax by distillation with steam, and recondensed. The crude wax differs widely according to the source, Thuringian lignites yielding a hard and brittle wax, whereas Bohemian lignites yield a softer product.

Ninety per cent of the montan wax present in the lignite is removed in this manner. About 10 to 15 per cent of the solvent is lost, but the high price obtainable for montan wax renders this permissible. Usually 8 to 10 per cent of montan wax is extracted from Thuringian lignite based on the dry weight of the latter. In exceptional cases, as high as 20 per cent has been obtained. Pyropissite yields between 50 and 70 per cent of montan wax based on its dry weight. Unfortunately, the supply of pyropissite is largely exhausted.

According to Edmund Graefe,[13] the following percentages of montan wax are extracted by benzol from the dried minerals:

	Per Cent
Bohemian Lignite	1.29
Texas Lignite	2.07
Lignite from the region of the Rhine	4.70
Lignite from Vladivostok	5.38
Lignite from Thuringia, Saxony	9.03
Pyropissite	69.50

The montan wax industry is not practiced in the United States, but is localized in Saxony.

Montan wax contains esters of acids possessing high molecular weight, free acids and a small quantity of substances containing sulfur. Various formulae have been assigned to it, including $C_{16}H_{32}O$, $C_{29}H_{58}O_2$, $C_{29}H_{58}O$, and $C_{42}H_{86}O$.

At ordinary temperatures it decomposes when distilled. It may, however, be purified by distilling with superheated steam *in vacuo*.[14] In this manner the following products are obtained:

(1) Pure odorless montan acid;
(2) Refined montan wax;
(3) A bright yellow wax containing paraffin;
(4) A residue containing paraffin;
(5) An acid-free pitch,[15] known as "montanpitch" or "montan-wax pitch," which is a hard waxy material having a dark color.

On heating with glycerine, an ester is obtained which has a much higher fusing-point (in the neighborhood of 200° F.). Montan wax may be blown with air, either alone or in the presence of oxalic acid,[16] and when chlorinated will be transformed into a resinous product.[17]

Commercial montan wax complies with the following characteristics:

(Test 1)	Color in mass:	
	Crude montan wax	Dark brown
	Product obtained by distillation in vacuo	Almost white
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Waxy
(Test 6)	Streak on porcelain	Yellowish brown to white
(Test 7)	Specific gravity at 77° F.	0.90–1.00
(Test 9*b*)	Penetration at 77° F.	0 to 5
(Test 9*c*)	Hardness at 77° F. (consistometer)	Above 100
(Test 9*d*)	Susceptibility index	> 100
(Test 10)	Ductility at 77° F.	0.0
(Test 15*f*)	Fusing-point (K. and S. method)	170–200° F.

Note.—Montan wax obtained from pyropissite has a higher fusing-point than that obtained from lignite; namely, between 190 and 200° F.

(Test 17)	Flash-point	550 to 575° F.
(Test 19)	Fixed carbon	2–10 per cent
(Test 21)	Solubility in carbon disulfide	Greater than 98 per cent
	Non-mineral matter insoluble	0–2 per cent
	Mineral matter	Less than 2 per cent
(Test 23)	Soluble in 88° petroleum naphtha	80–100 per cent
(Test 26)	Carbon	82–83½ per cent
(Test 27)	Hydrogen	14–14½ per cent
(Test 28)	Sulfur	Less than 1.5 per cent
(Test 29)	Nitrogen	Trace
(Test 30)	Oxygen	3–6 per cent
(Test 33)	Solid paraffins	0–10 per cent
(Test 34*b*)	Sulfonation residue	0–10 per cent
(Test 37*a*)	Acid value	28–33
(Test 37*c*)	Ester value	28–73

(Test 37*d*)	Saponification value	50–95
(Test 37*e*)	Saponifiable matter	50–80 per cent
	Resins	20–30 per cent
	Alcohols	5–20 per cent
	Normal wax acids	49–52½ per cent
	Oxy-acids	3–5 per cent
	Sulfur-containing acids (characteristic of crude montan wax)	6½–8 per cent
(Test 38)	Asphaltic constituents	0 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Montan wax is used for manufacturing shoe polishes, phonographic records, electrical insulating materials, and the like, also for impregnating timber under vacuum followed by pressure.[18]

CHAPTER VIII

NATIVE ASPHALTS OCCURRING IN A FAIRLY PURE STATE

Under this heading will be considered the most important asphalt deposits containing less than 10 per cent of mineral matter figured on the dry weight. These include exudations or seepages of liquid or semi-liquid asphalts, also surface overflows and lakes. Most of these are characterized by being liquid to semi-liquid at normal atmospheric temperature, and by containing a comparatively large proportion of volatile matter. Only a few of these deposits are of value commercially.

The principal deposits are as follows:

NORTH AMERICA

UNITED STATES

Kentucky.

Breckinridge County. The so-called "Tar Springs" situated about 4 miles south of Cloverport on Tar Creek, have been known for many years. They occur as seepages of pure, soft asphalt at the base of an overhanging cliff of sandstone where it joins a stratum of limestone. The asphalt is accompanied by water charged with sulfur compounds, and the surrounding rocks abound in marine fossils.

Grayson County. Similar seepages abound along Big Clifty Creek and its tributaries, in the vicinity of Grayson Springs station. Some exude from sandstone and others from clay or shale. The seepages carry between 5 and 10 per cent of free mineral matter, the balance consisting of a very soft, "stringy" asphalt containing a large proportion of volatile ingredients and yielding about 15 per cent of fixed carbon.

Oklahoma.

There are only a few minor occurrences of pure asphalt found in Oklahoma in the form of seepages, including the following:

Carter County. NE ¼, Sec. 10, T 2 S, R 2 W; 10 miles north of Wheeler.

Murray County. SW ¼, SE ¼, Sec. 15, T 1 S, R 3 E; 3 miles south of Sulphur.

Neither of these has any commercial importance.

Utah.

Uinta County. A pure, solid asphalt is found in Tabby Canyon, a branch of the Duchesne River, 8 to 9 miles south and west of the town of Theodore and about 30 miles west of Ft. Duchesne. It has been exploited under the name "tabbyite" [1] and tests as follows:

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.006–1.010
(Test 9*a*)	Hardness, Moh's scale	Less than 1
(Test 9*b*)	Penetration at 77° F	0
(Test 9*c*)	Consistency at 77° F	80.0
(Test 15*a*)	Fusing-point (K. and S. method)	178° F.
(Test 16)	Volatile matter, 325° F. in 5 hrs	2.78 per cent
	Volatile matter, 400° F. in 5 hrs	6.40 per cent
(Test 19)	Fixed carbon	8.08– 9.2 per cent
(Test 21)	Soluble in carbon disulfide	94.7 –92.1 per cent
	Non-mineral matter insoluble	0.5 – 1.1 per cent
	Free mineral matter	4.8 – 6.8 per cent
(Test 22)	Carbenes	0.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	61 per cent
(Test 25)	Carbon	82 per cent
(Test 26)	Hydrogen	11 per cent
(Test 27)	Sulfur	2 per cent
(Test 28)	Nitrogen	2–2.5 per cent
	Undetermined	2 per cent

Boxelder County. A pure viscous asphalt deposit occurs below the bed of Great Salt Lake, about 10 miles south of Rozel, in the Promontory Range.[2] It is found in a series of horizontal veins 3 to 5 ft. thick interposed between beds of clay, continuing to a depth of at least 140 ft. It is highly probable that a lake of asphalt occurred at this point centuries ago, which in time became covered with sediments, giving rise to a series of veins.

At the present time, masses of asphalt exude through the unconsolidated material at the bottom of the lake, and float to the surface in lumps 1 to 2 ft. in diameter. This occurrence corresponds very closely with the Dead Sea deposit. On analysis, the asphalt tests as follows:

(Test 5)	Lustre	Very bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.02
(Test 9*b*)	Penetration at 32° F. (200 g. in 60 secs.)	12
	Penetration at 77° F. (100 g. in 5 secs.)	50
	Penetration at 115° F. (50 g. in 5 secs.)	170
(Test 10*a*)	Ductility at 77° F.	70 cms.
(Test 16)	Volatile at 325° F. in 5 hrs.	2.33 per cent
	Volatile at 325° F. in 7 hrs.	3.4 per cent
(Test 19)	Fixed carbon	4.56 per cent
(Test 21)	Soluble in carbon disulfide	95.00 per cent
	Non-mineral matter insoluble	1.84 per cent
	Free mineral matter	3.16 per cent

Note.—Has unusually high adhesiveness, ductility and cementing qualities.

California.

Kern County. Deposits of asphalt occur in the so-called "Asphalto Region" in the western part of Kern County, about 50 miles west of Bakersfield, in the form of large springs; also as veins. The character of the asphalt varies greatly, both in consistency and purity. The superficial overflow covers an area of 7 acres, in a layer 2 to 4 ft. thick overlying sand and clay. Part of it has hardened on account of exposure to the elements, and other portions are still soft and viscous. A vein of asphalt also occurs in the vicinity of the overflow, filling a fault, varying from 2 to 8 ft. in width, averaging about 4 ft. The nature of the asphalt in the vein is similar to that of the overflow.

The asphalt carries from 3 to 30 per cent mineral matter, mostly sand and clay, also gas, which is evidenced by the fact that it loses between 5 and 15 per cent in weight on being heated to 212° F. for one hour. The run of the mine averages 85 per cent asphalt, 10 per cent mineral matter and 5 per cent moisture and gas. It is refined by heating, which drives off the water and gas and permits a certain amount of the mineral matter to settle out. According to Clifford Richardson[8] the refined asphalt tests as follows:

(Test 4)	Fracture	Semi-conchoidal
(Test 5)	Lustre	Bright to dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.06
(Test 9*b*)	Penetration at 77° F.	0–27
(Test 16)	Volatile matter, 325° F., 5 hrs.	6.6 per cent
	Volatile matter, 400° F., 5 hrs.	19.9 per cent
(Test 19)	Fixed carbon	8.0 per cent
(Test 21)	Soluble in carbon disulfide	89.8 per cent
	Non-mineral matter insoluble	3.4 per cent
	Free mineral matter	6.8 per cent
	Total	100.0 per cent
(Test 22)	Carbenes	0.3 per cent
(Test 23)	Soluble in 88° petroleum naphtha	53.4 per cent
(Test 34*a*)	Saturated hydrocarbons	28.6 per cent

The asphalt resembles gilsonite in its outward appearance, but is considerably softer, yielding a smaller percentage of fixed carbon. Richardson infers that the asphalt has been metamorphized only part way to gilsonite. This deposit of asphalt is not being worked at the present time, but is of interest from the scientific viewpoint.

A sample of liquid asphalt taken from seepages in the so-called "McKittrick Region," in Kern County, shows specific gravity at 77° F. of 0.99, and 16 per cent loss on being heated to 400° F. for seven hours. In its original state it is very soft and sticky.

San Luis Obispo County. A large surface deposit of soft asphalt produced by seepage from the surrounding shale occurs at Tar Spring Creek, a tributary of the Arroyo Grande, 20 miles southeast of San Luis Obispo, covering an area of 200 ft. in diameter and 3–15 ft. deep. As it exudes from the shale, the asphalt is soft and accompanied with sulfurous water; near the edge of the deposit it appears quite hard, and at the edge it verges towards brittleness. No analytical results are available.

Santa Barbara County. Veins of high-grade asphalt occur in La Graciosa hills about 4 to 5 miles east of the town of Graciosa in the so-called Santa Maria Region. These are irregular in formation, extending through shale and sandstone, and varying from several inches to 2 ft. in width. Associated with these veins are beds of impregnated asphaltic shale, extending over an area of a mile or two, and containing a variable percentage of asphalt. One of the striking features of these occurrences is the presence of marine fossils in the veins and surrounding shale, indicating that the asphalt is of animal origin.

Oregon.

Coos County. An unusual type of asphalt occurring in beds of coal has been reported at the old Newport Mine at Libby, and old Ferrey's Mine at Riverton, in the Coos Bay coal field. It is hard and brittle, and similar to coal in appearance. About one-third of the non-mineral matter is insoluble in carbon disulfide, yet the material fuses as a comparatively low temperature (about 300° F.), and has a specific gravity of less than 1.10 at 77° F. It may be regarded as a metamorphized asphalt or a glance pitch. It constitutes one of those substances encountered occasionally, falling on the border line, so that it becomes a difficult matter to arrive at its correct classification. For a long time it was known as a "Pitch Coal." [4] The following data would seem to indicate that it partakes of the properties of an asphalt rather than of a glance pitch:

(Test 4)	Fracture	Hackly to conchoidal
(Test 5)	Lustre	Fairly dull to brilliant
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.09 to 1.28
(Test 9*a*)	Hardness, Moh's Scale	About 1
(Test 9*b*)	Penetration at 77° F	0
(Test 9*c*)	Consistency at 77° F	Above 100
(Test 14*b*)	In flame	Softens and flows
(Test 15*a*)	Fusing-point (K. and S. method)	302 to 330° F.
(Test 16)	Volatile at 325° F., 5 hrs	0.5 per cent
(Test 19)	Fixed carbon	10–13 per cent
(Test 21)	Soluble in carbon disulfide	100 per cent
(Test 22)	Carbenes	0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	About 10 per cent
(Test 27)	Sulfur	0.5–1.0 per cent

Mexico

State of Tamaulipas.

Asphalt springs occur at numerous points along the Tamesi River, which, according to Clifford Richardson,[5] show the following characteristics:

(Test 7)	Specific gravity at 77° F	1.04–1.12	
(Test 9*b*)	Penetration at 77° F	40–16	
(Test 16)	Loss at 212° F. until dry	10–20	per cent
	Loss at 325° F. for 5 hrs. (refined material)	1.5–4.8	per cent
	Loss at 400° F. for 5 hrs	4.3–8.9	per cent
(Test 17)	Flash-point	308° F.	
(Test 19)	Fixed carbon	12.6–16.1	per cent
(Test 21)	Solubility in carbon disulfide (refined material)	89.0–99.0	per cent
	Non-mineral matter insoluble	0.5– 1.8	per cent
	Free mineral matter	0.5– 9.1	per cent

Other deposits in the neighborhood show a larger proportion of mineral matter, often running as high as 33 per cent.

Chijol. Asphalt springs occur also near Chijol, 25 miles west of Tampico. They are comparatively soft in consistency, testing over 90 per cent soluble in carbon disulfide, with less than 10 per cent mineral matter.

State of Vera Cruz.

District of Tuxpan. Similar deposits are found in the neighborhood of Tuxpan, some distance from the Tuxpan River, having the same general characteristics as the preceding. Analyses show that 90 per cent is soluble in carbon disulfide, with less than 10 per cent of mineral matter.

District of Chapapote. Similar deposits are found 15 miles from Timberdar at the head of the Tuxpan River, of an exceedingly pure character, testing 99 per cent soluble in carbon disulfide, and less than 1 per cent mineral matter. The asphalt varies in consistency from a semi-liquid to a comparatively hard solid, depending upon the length of time it has been exposed to the weather.[6]

In the early days, the name "chapapote" was applied generally to designate viscous, semi-liquid asphalts corresponding in characteristics to the type of asphalt found in this district.

CUBA

Province of Matanzas. A pit filled with pure liquid asphalt has been reported in the neighborhood of Santa Catalina. This occurs in a bed of serpentine, and originally produced in the neighborhood of 20 barrels of semi-liquid asphalt a day, derived presumably from underlying petroleum-bearing strata. Other pits in the neighborhood similarly yield liquid asphalt.

Liquid asphalt is also found near Cardenas and extends some distance eastwards. Another deposit, known as "Dos Companeros Mine," occurs in the Guametos District, near Sabanilla de la Palma, testing as follows:

(Test 7)	Specific gravity at 77° F.	1.05–1.06
(Test 16)	Volatile at 220° F.	2–18 per cent
	Volatile at 325° F., 5 hrs.	2.8–6.8 per cent

(Test 19)	Fixed carbon	10–12 per cent
(Test 21)	Soluble in carbon disulfide	98–99 per cent
	Non-mineral matter insoluble	0–1.5 per cent
	Mineral matter	0.5–2.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	73–77 per cent

Another deposit occurs 7 miles south of Hato Nuevo, testing similar to the preceding. All of these deposits contain more or less adventitious water.

Province of Santa Clara. A variety of hard asphalt, approaching glance pitch, is found in Calabazar District, near the town of Mata, in a mine known as "El Provenir." It tests as follows:[7]

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak on porcelain	Brownish black
(Test 7)	Specific gravity at 77° F.	1.09–1.11
(Test 19)	Fixed carbon	18.0– 19.2 per cent
(Test 21)	Soluble in carbon disulfide	97.5– 97.6 per cent
	Non-mineral matter insoluble	0.5– 0.9 per cent
	Mineral matter	2.0– 1.5 per cent
	Total	100.0–100.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	39.4– 57.4 per cent

Province of Camaguey. Deposits of pure soft asphalt occur near the village of Minas, about 30 miles from Nuevitas and 20 miles from Puerto Principe City. The asphalt is associated with water, and tests as follows:

(Test 7)	Specific gravity at 77° F. (dry)	1.079
(Test 9*b*)	Hardness at 77° F. (dry)	35
(Test 16)	Volatile at 325° F. in 5 hrs	6.2 per cent
	Penetration of residue at 77° F.	12
(Test 21)	Soluble in carbon disulfide	94.5 per cent
	Non-mineral matter insoluble	0.3 per cent
	Mineral matter	5.2 per cent
	Total	100.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	70.0 per cent

Province of Santiago de Cuba. A small deposit of soft asphalt has been reported in Victoria de las Tunas District, 3½ miles

southwest of San Manuel, in a mine known as "Punta la Brea," which tests as follows:

(Test 7)	Specific gravity at 77° F	0.990
(Test 16)	Volatile at 325° F. in 5 hrs	6.3 per cent
(Test 21)	Soluble in carbon disulfide	99.1 per cent
	Non-mineral matter insoluble	0.5 per cent
	Mineral matter	0.4 per cent
(Test 23)	Soluble in 88° petroleum naphtha	88.8 per cent

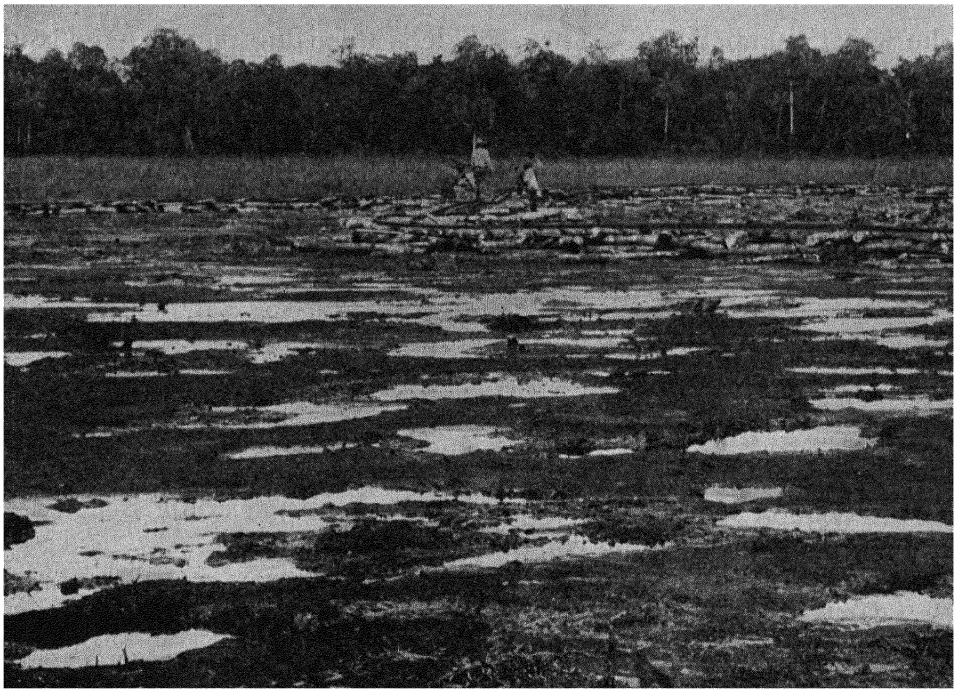
SOUTH AMERICA

VENEZUELA

State of Bermudez. The so-called Bermudez "Pitch Lake" [8] is situated near the town of Guanoco in the District of Benitez, 3 miles above the confluence of the San Juan and Guanoco Rivers, 25 miles west of the Gulf of Paria, and 105 miles due west of Trinidad Lake. It is covered with vegetation and water in pools. A typical view is shown in Fig. 43. The lake represents the exudation of soft asphalt from springs distributed at different points over its area, and constitutes one of the largest deposits of pure asphalt yet discovered, extending over 1110 acres and varying in depth from 2 to 20 ft., averaging 5 ft.

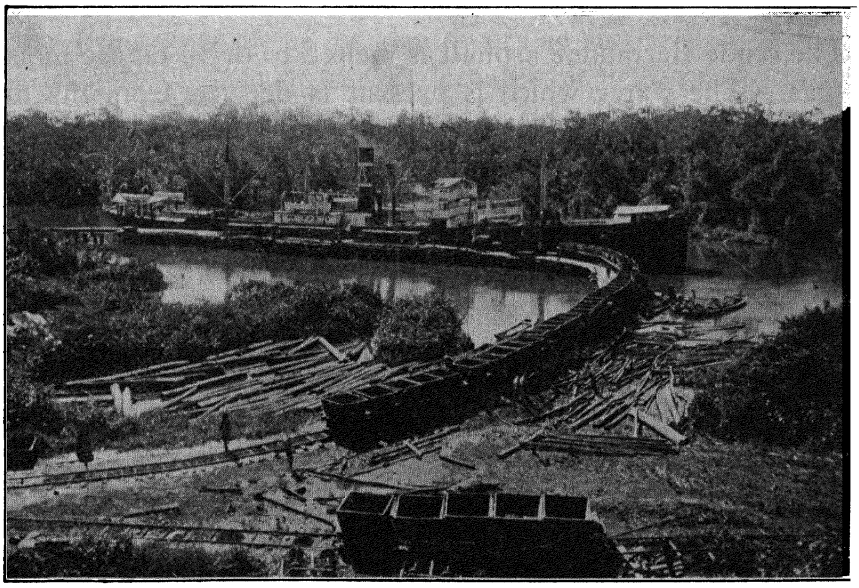
Its consistency varies in different parts of the lake. Where it exudes from the springs, it is quite soft, and disengages gas freely and copiously. The surface of the deposit hardens slowly on exposure to the weather, forming a crust varying from several inches to several feet in thickness, and sufficiently firm to support the weight of a man. The asphalt underneath, however, is still soft and semi-liquid, and there are numerous breaks through the surface from which the soft asphalt oozes. At the edge of the lake the asphalt is hard and brittle, due to the evaporation of the volatile constituents by the heat of the sun. Certain portions of the lake have been converted into a cokey mass as a result of fires which must have swept over the lake years ago, due probably to the combustion of vegetation growing profusely at the edges.

To remove the asphalt, a dam is built of slag and waste and



Courtesy of Barber Co.

FIG. 43.—View of Bermudez Asphalt Lake.



Courtesy of Barber Co.

FIG. 44.—Transporting Bermudez Asphalt.

the water pumped out. The asphalt is then dug out by hand and loaded into small cars, which are hauled by cable to a railroad running to Guanoco, 8 miles distant, where it is either refined or run directly to a loading jetty, whence it is transferred to steamers as shown in Fig. 44.

The crude asphalt has the following composition:[9]

(Test 21)	Soluble in carbon disulfide	64.39 per cent
	Non-mineral matter insoluble	3.53 per cent
	Mineral matter	2.08 per cent
(Test 25)	Water	30.00 per cent
	Total	100.00 per cent

According to Clifford Richardson,[10] the dried crude Bermudez asphalt has the following composition:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 7)	Specific gravity at 77° F.	1.05–1.075
(Test 16)	Volatile at 400° F. in 5 hrs. (dried material)	5.81–16.05 per cent
(Test 21)	Soluble in carbon disulfide	90–98 per cent
	Non-mineral matter insoluble	0.62–6.45 per cent
	Free mineral matter	0.50–3.65 per cent

The crude Bermudez asphalt is melted to drive off the moisture and gas. The water which is present is derived from the heavy rains and by overflows from the surrounding country. It is *not* emulsified with the asphalt as is the case with the Trinidad deposit. The percentage of water varies from 10 to about 40 per cent as a maximum.

Refined Bermudez asphalt tests as follows:[11]

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Very bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.06–1.085
(Test 9*a*)	Hardness on Moh's scale	Less than 1
(Test 9*b*)	Penetration at 115° F.	60
	Penetration at 77° F.	20–30
	Penetration at 32° F.	3
(Test 9*c*)	Consistency at 115° F.	7.7
	Consistency at 77° F.	22.7
	Consistency at 32° F.	93.8
(Test 9*d*)	Susceptibility index	62.5

(Test 10*b*)	Ductility at 115° F.	14.5
	Ductility at 77° F.	11
	Ductility at 32° F.	0
(Test 11)	Tensile strength at 115° F.	0.60
	Tensile strength at 77° F.	3.45
	Tensile strength at 32° F.	10.5
(Test 15*a*)	Fusing-point (K. and S. method)	130–140° F.
(Test 15*b*)	Fusing-point (R. and B. method)	145–160° F.
(Test 16)	Volatile matter, 325° F., 5 hrs.	3.0– 6.0 per cent
	Volatile matter, 500° F., 5 hrs.	8.0–10.0 per cent
(Test 19)	Fixed carbon	12.9–14.0 per cent
(Test 21)	Solubility in carbon disulfide	92–97 per cent
	Non-mineral matter insoluble	1.5–4.0 per cent
	Free mineral matter	1.5–6.5 per cent
(Test 22)	Carbenes	0.0–1.0 per cent
(Test 23)	Solubility in 88° petroleum naphtha	60–75 per cent
(Test 26)	Carbon	82.88 per cent
(Test 27)	Hydrogen	10.79 per cent
(Test 28)	Sulfur	5.87 per cent
(Test 29)	Nitrogen	0.75 per cent
	Total	100.29 per cent
(Test 33)	Solid paraffins	Trace
(Test 34*a*)	Saturated hydrocarbons	23–25 per cent
(Test 37*d*)	Saponification value	28.0 per cent
(Test 38*a*)	Free asphaltous acids	3.5 per cent
(Test 38*b*)	Asphaltous anhydrides	2.0 per cent
(Test 38*c*)	Asphaltenes	35.3 per cent
(Test 38*d*)	Asphaltic resins	14.4 per cent
(Test 38*e*)	Oily constituents	39.6 per cent

La Brea Deposit. This also occurs as an overflow in the form of a lake at La Brea, in the Pedernales oil field, on the northwest coast of the Island of Capuré, in the delta of the Orinoco River, some distance east of the Bermudez Lake. It is about 3200 ft. long and 100 to 200 yards wide, and is fed by several springs and one asphalt cone. Similar deposits on a smaller scale are found on the neighboring islands of Paquero and Del Plata, likewise in the District of Sucre, where cones of asphalt occur 5 ft. high and 30 ft. wide.

State of Zulia.

Maracaibo Deposit. A number of asphalt deposits occur in the Inciarte region, in the District of Mara, south of the Limon River, 60 miles west of the city of Maracaibo. Others occur at La Paz, a short distance to the east. These are in the form of overflows, exuding from springs. More than 100,000 tons were collected

during 1901–1905, but operations have since largely ceased. The asphalt was gathered by means of picks and shovels and transported in barges down the Limon River to the island Toas at the head of the Gulf of Maracaibo, where it was loaded on board steamers. It melts at a higher temperature than the Bermudez asphalt, and possesses a very strong and characteristic sulfurous odor.

According to Clifford Richardson [12] it tests as follows:

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Very bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.06–1.08
(Test 9*a*)	Hardness, Moh's scale	Less than 1
(Test 9*b*)	Penetration at 77° F.	20–30
(Test 16)	Volatile at 325° F., 5 hrs.	1.5–6 per cent
	Volatile at 500° F., 5 hrs.	4.7– 6.0 per cent
(Test 19)	Fixed carbon	15.0–19.0 per cent
(Test 21)	Solubility in carbon disulfide	92–97 per cent
	Non-mineral matter insoluble	1.4–5.0 per cent
	Free mineral matter	1.5–6.0 per cent
(Test 22)	Carbenes	1.5 per cent
(Test 23)	Solubility in 88° petroleum naphtha	45–55 per cent
(Test 34)	Saturated hydrocarbons	25–30 per cent

EUROPE

FRANCE

Department of Puy-de-Dôme (Auvergne). In the vicinity of Clermont-Ferrand, seepages of soft asphalt exude from crevices in the rock, containing 90 per cent of asphalt, 7 per cent water, and 3 per cent mineral substances. The exudations are comparatively small in amount, and the asphalt has never proved of importance commercially.

At Pont-du-Château there occur extensive deposits of rock asphalt, from which soft asphalt seeps in moderately large quantities and is caught in subsidiary workings excavated for this purpose. Prior to 1914, approximately 100 tons of pure liquid asphalt were recovered annually in this manner. This represents practically the only locality in Europe where an appreciable quantity of pure asphalt is obtained at the mines.

ALBANIA

Sélénitza (Sélinitza). At the junction of the Vojutza (Vojusa) and Sauchista Rivers, extending from the towns of Kanina to Berat, in the vicinity of the Bay of Avlona (Valona), there occurs a fairly large deposit of moderately hard asphalt in sandstone and conglomerate, in veins as wide as 10 ft. Marine fossils are associated with this deposit, indicating it to be of animal origin. The asphalt breaks with a conchoidal fracture, showing a high lustre. It contains between 8 and 14 per cent of mineral matter, averaging about 10 per cent. Comparatively large quantities have been mined during many centuries.

GREECE

Zante. An extensive deposit of asphalt occurs in the southern portion of the Island of Zante, in the form of springs and seepages. The asphalt is very soft in consistency, having a specific gravity of 1.00 to 1.02 at 77° F., and carrying but a trace of mineral matter, with a fairly large proportion of water in emulsion. The springs occur in a region of clay and limestone, more or less saturated with petroleum. These deposits have been worked for many generations. The asphalt is refined in a crude way by the natives who use it for calking the seams of ships, and as a mortar for cementing together the stones of buildings, following the same method as practiced centuries ago.

RUSSIA [13]

Kutais Province (Transcaucasia). In the neighborhood of Osurgeti, about 3 km. north of the railroad station Notanebi and about 400 m. from the railway (near the villages Yakobi and Narudcha), there occurs a vein of fairly pure asphalt containing 9.66 per cent ash. Similarly, at Sakuprisgale brook, near the village of Sameho, about 4 km. from the railroad station Notanebi, there is found a deposit of pure hard asphalt containing 4.98 per cent ash.

Tiflis Province (Transcaucasia). In the vicinity of Tiflis, on the right bank of the Yora River, southwest of the Cloister of Holy David of Garedchi, there occurs a deposit of hard asphalt containing 3.86 per cent ash.

Uralsk Province (Russia in Asia). In the so-called Kirgisen Steppes, near where the rivers Emba and Sagis empty into the Caspian Sea, there occur asphalt deposits at Imankara and Karamurat; also about 4 km. east of Alascha-Kasgen there are found two deposits of pure asphalt, one covering 4300 sq. m. and the other 1600 sq. m., ranging from a hard to a softer material of elastic consistency.

ASIA

SYRIA (LEVANT STATES)

Villayet of Beirut.[14] At Jebel Keferie (Kfarieh), a hill near the bend of the Nahr el Kebir, about 40 km. from the sea, on the road between Latakia and Aleppo, there occurs a large deposit of asphalt, covering an area 1400 by 1500 m., part of which runs quite free from mineral contamination (contains as low as ½ per cent ash). It has not been worked to any extent, on account of the difficulty in transportation to the coast.

MESOPOTAMIA (IRAQ)

Deposits of pure asphalt have been reported at the following places:

Hit. Containing 64 per cent asphalt and 36 per cent water. The extracted asphalt contained 0.5 per cent ash and 8.3 sulfur, fusing-point (R. and B.) 47.5° C., penetration at 77° F. 108.

Ain el Maraj (near Kerkuk). Containing 79 per cent asphalt and 21 per cent water. The extracted asphalt contained 3.8 per cent ash and 8.8 per cent sulfur, fusing-point (R. and B.) 64° C., penetration at 77° F. 25.

Ain Ma' Moura (*Ain Mamurah*). Containing 72 per cent asphalt and 28 per cent water. The extracted asphalt contained 0.7 per cent ash and 8.5 per cent sulfur, fusing-point (R. and B.) 52.5° C., penetration at 77° F. 73.

Quijarah, Ramadi and Abu Gir. Minor asphalt seepages have been noted.

ASIATIC RUSSIA

Sakhalin.

Province of Nutowo. An asphalt lake, known as the "Great Okha Asphalt Lake," occurs in Okha, on the east coast of the Island of Sakhalin in a swampy valley, associated with a very thick variety

of petroleum, exuding in the neighborhood. Where the asphalt emanates from the springs, it is very soft and sticky, but towards the edges of the lake it is hard and brittle. The asphalt has a rather strong odor, and contains a substantial quantity of volatile matter. As mined, it contains: asphalt 89.7 per cent, ash 1.18 per cent and water 9.03 per cent, and has a fusing-point of 73° C. (K. and S.) and a penetration at 77° F. of 17. This asphalt occupies an intermediate position between Trinidad and Bermudez Lake asphalts.[15] After being air-dried, it carries 0.75 per cent of moisture, 0.22 per cent of ash, and the balance pure asphalt containing 0.80 to 0.85 per cent sulfur. It is estimated that at least 400,000 tons of asphalt, averaging 0.9 per cent of mineral matter, are present in the lake. Up to the present, the deposit has not been developed commercially.[16]

Philippine Islands

Island of Leyte.

Several asphalt deposits have been found in this region, one near the head of the Butason River, about 6 miles from the Barrio of Campocpoc, on the northwestern coast of the island, and another near the town of Villaba.[17] These occur in limestone and sandstone, and extend over an area 12 miles long. Outcrops of various grades of asphalt have been reported, including the solid, viscous and liquid types. Both pure and rock asphalts are found, the latter carrying a variable proportion of sand. Two varieties of pure, hard asphalt were examined by the author, one having a black color in mass, and a glossy, black, conchoidal fracture: another having a dark brown color in mass, with a hackly, dull fracture. They tested as follows:

		Black Asphalt	*Brown Asphalt*
(Test 4)	Fracture	Conchoidal	Hackly
(Test 5)	Lustre	Bright	Dull
(Test 6)	Streak on porcelain	Black	Yellowish brown
(Test 7)	Specific gravity at 77° F.	———	0.978
(Test 9*b*)	Penetration at 77° F.	10	2
(Test 9*c*)	Consistency at 77° F.	31.7	Above 100
(Test 10*a*)	Ductility at 77° F.	½	0
(Test 15*a*)	Fusing-point (K. and S. method)	287° F.	138° F.
(Test 16)	Volatile at 500° F., 5 hrs.	3 per cent	1.15 per cent
(Test 19)	Fixed carbon	10.5 per cent	9.4 per cent
(Test 21)	Soluble in carbon disulfide	98 per cent	99 per cent
	Mineral matter	2 per cent	1 per cent
(Test 37*e*)	Saponifiable matter	0 per cent	0 per cent

The brown variety is unique. It is somewhat similar in physical properties to ozokerite, but it is very much more friable. When melted it turns black in mass, becoming lustrous (although it still shows a yellowish brown streak). It has been marketed under the name "leyteite." The black asphalt is not classed as an asphaltite in view of its comparative softness at 77° F. These deposits have not yet been exploited commercially.

CHAPTER IX

NATIVE ASPHALTS ASSOCIATED WITH MINERAL MATTER

This chapter will include the principal deposits of natural asphalts containing 10 per cent and over of associated mineral matter, based on the dry weight. Such deposits include veins, strata and lake formations.

NORTH AMERICA

UNITED STATES [1]

Kentucky.

All the deposits in the State of Kentucky are composed of sand and sandstone, carrying between 4 and 12 per cent of soft asphalt filling the interstices.[2] These deposits occur in strata from 5 to 40 ft. thick, along the eastern and southeastern border of the coal field in Hardin, Grayson, Breckinridge, Edmonson, Hart, Warren, Butler, Logan, and other adjoining counties.

Hardin County. A deposit of sand asphalt occurs a few miles northeast of Summit, in the southwestern portion of Hardin County.

Carter County. This deposit occurs one-half mile southeast of the town of Soldier, and consists of unconsolidated quartz grains held together by 4 to 10 per cent of asphalt, which is comparatively soft and contains a goodly proportion of volatile matter.

Breckinridge County. This deposit is located from 2 to 4 miles south of Garfield, and is composed of unconsolidated quartz grains carrying 4 to 8 per cent of asphalt. It forms a hillside ledge about 14 ft. thick with an overburden of 10 to 20 ft. The deposit has not been worked to any great extent in recent years, although formerly it was of considerable interest in the paving industry. Other prospects occur in this neighborhood, but these have not been developed.

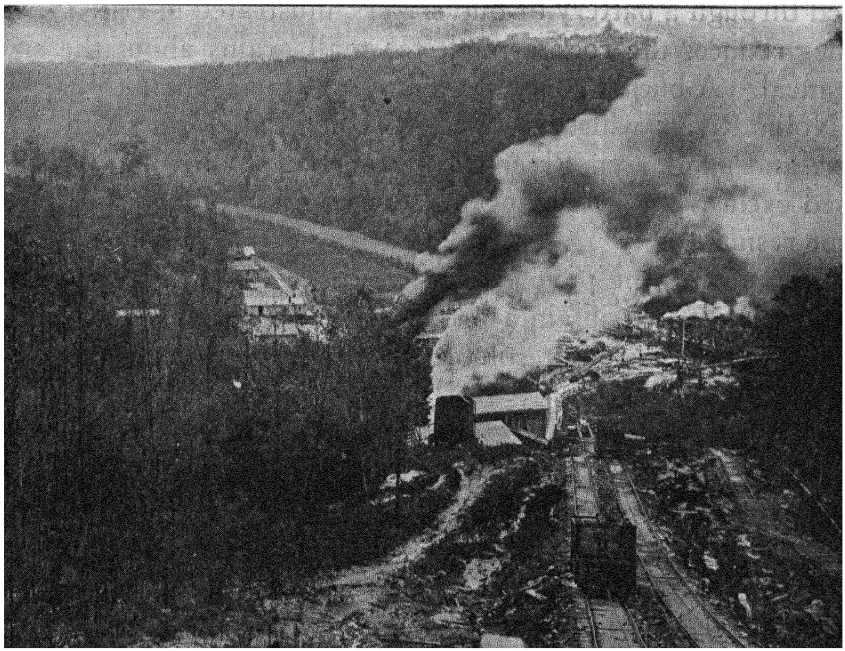
Grayson County. Two deposits have been worked in this locality in the vicinity of Big Clifty and Grayson Springs Station, one

3 miles southwest, and the other 9 miles north of Leitchfield. The former occurs in a stratum 5 ft. thick, impregnated with 6 per cent of asphalt, in an unconsolidated quartz sand. The second was formerly one of the most active mines in Kentucky, but has now been idle for a number of years. It consists of a stratum 10 ft. thick, carrying 7–12 per cent of very soft asphalt. A number of seepages are in evidence along the side walls of the quarry and since the asphalt contains a large proportion of volatile matter, they soon harden on exposure to the weather. Some of the seepages examined by Richardson contained 30 to 65 per cent of mineral matter, the extracted asphalt showing a penetration of between 35 and 45 at 77° F., and yielded 12 per cent of fixed carbon. Another deposit occurs at Tar Hill.

Edmonson County. Deposits of asphaltic sandstone, estimated to exceed 200 million tons, covering over 7000 acres, are located about 6 miles due west of Brownsville and about 12 miles northeast of Bowling Green. The properties adjoin both Bear Creek and Green River, being located on both sides of the former and north of the latter. Soil overburden of 5 to 40 ft. covers a horizontal stratum of rock asphalt 15 to 30 ft. thick. The latter analyzes as follows:

(Test 21)	Soluble in carbon disulfide.....	4.22–	9.00 per cent
	Matter insoluble.....	95.64–	90.91 per cent
(Test 25)	Moisture at 105° C.....	0.14–	0.09 per cent
		100.00	100.00 per cent

At the present time the only deposits in the State of Kentucky worked to any extent occur at Kyrock and at the town of Asphalt, on the Nolin River, about 10 miles west of the celebrated Mammoth Cave, near Brownsville. They consist of a stratum of fine sand impregnated with 8 to 10 per cent of asphalt, occurring in irregular beds 5 to 36 ft. thick in a horizontal stratum below an overburden of clay and unimpregnated sandstone, 6 to 30 ft. thick. The overburden is removed by a hydraulic pressure line, and where it does not yield to this, a steam shovel is used. The rock asphalt is removed by quarrying as illustrated in Figs. 45 and 46, and conveyed by a gravity tramway to a crusher which reduces the rock from 5- to 10-in. fragments to lumps about the size of an egg. It is then



Courtesy Kentucky Rock Asphalt Co.

FIG. 45.—Asphalt Deposit at Kyrock, Kentucky.

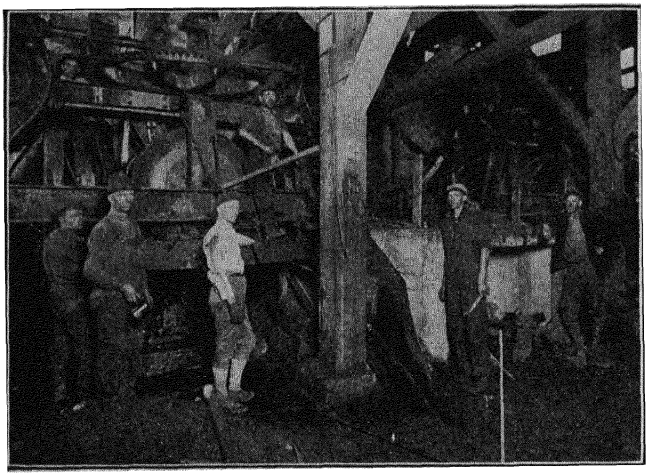


Courtesy Kentucky Rock Asphalt Co.

FIG. 46.—Hillside Quarry of Kyrock Asphalt Deposit.

passed through a battery of pulverizers, illustrated in Fig. 47, which further reduces the rock asphalt to small grains about the size of corn meal. A bucket conveyor transports the asphalt to barges on the Nolin River, which in turn are conveyed down to the Green River, and up Barren River to the railroad at Bowling Green. It is used exclusively for paving purposes, and is reported to give excellent results.

In preparing the paving mixture, rocks of varying asphalt content are blended to obtain a product carrying uniformly 7 per cent ($\pm$ ¼ per cent) asphalt soluble in carbon disulfide. In applying



Courtesy Kentucky Rock Asphalt Co.

FIG. 47.—Pulverizing Kentucky Rock Asphalt at Kyrock, Kentucky.

the asphalt, it is spread loosely over the primed base in a layer about 3 in. thick over macadam (9 to 10 in. thick), or a concrete base (6 in. thick), and compacted *cold* with a 15-ton steam roller, to a thickness of 2 in., provided, however, that the air temperature is not lower than 60° F. If necessary, the surface may be planed after the first rolling, by which the excess material is shaved from the high places and deposited in the depressions. The product has the advantages of not requiring a hot mixing plant. It presents a rather gritty surface, which prevents slipping, does not become wavy under traffic, and may be readily repaired. To offset these, it has the disadvantage of being marked by traffic for several days after being laid, and moreover it is claimed that the drip from

autos readily washes out the asphalt content, leaving unconsolidated sand.

Warren County. Several deposits of sand asphalt are located at Youngs Ferry on the Green River, 12 miles north of the town of Bowling Green. One occurs in a bed about 10 ft. thick, and carries between 6 and 9 per cent of asphalt. A second consists of a vein 5 to 15 ft. thick containing about the same percentage of asphalt. Both are undeveloped. The extracted asphalt shows a penetration at 77° F. of 200, and much volatile matter (13 per cent at 400° F. in seven hours).

Logan County. A quarry has been opened up about 5 miles northeast of Russellville, exposing about 15 ft. of asphaltic sandstone in a bed about 100 ft. long. The rock carries about 7 per cent of asphalt, which shows very much less volatile matter than the preceding (about 4 per cent loss at 325° F. in five hours). This mine is no longer active.

Missouri.[3]

Lafayette County. A bed of asphaltic sand occurs 1½ miles northwest of Higginsville, carrying 8½ per cent of asphalt, associated with sandy shale. This deposit has not been worked commercially.

Indiana.

While drilling for oil at Princeton, a bed of asphalt several feet thick was found 100 ft. below a vein of coal. Seepages of liquid asphalt have also been reported in a well in the neighborhood. None of these have been developed.[4]

Oklahoma.

This state is one of the richest asphalt-bearing centers in the United States. Asphalts are found in both the liquid and solid forms, occurring as springs, seepages and rock impregnations. Practically all the deposits are found in the southern portion of the state, between the 35th parallel of north latitude, and the Red River on the south, and included between the Arkansas line on the east, to the city of Granite, Oklahoma, on the west. This area is shown in Fig. 48, and includes deposits or prospects in the following counties: Comanche, Jefferson, Stephens, Garvin, Carter, Murray, Love, Marshall, Johnston, Pontotoc, Atoka, McCurtain and Leflore.

TABLE XVI

Rock Asphalts in Oklahoma

Base	Locality (S = Section, T = Township, R = Range)	Nearest City or Town	Remarks
	Comanche County Wichita Mountain Region		
Sand	Sec. 26, T 4 N, R 11 W	3 to 4 m. W of Elgin	Exploration pit; material used for pavements of Lawton, Okla.
Sand	NE ¼, Sec. 24, T 2 N, R 11 W	6 m. NE of Lawton	Small outcrop with oil seepage. Undeveloped.
Sand	W ½, NW ¼, Sec. 21, T 2 N, R 12 W	6 m. NW of Lawton	Low asphalt content. Along a fault. Exploited considerably.
Sand	SE ¼, Sec. 29, T 5 N, R 12 W	8 m. SW of Apache	Undeveloped deposit in the Permian Redbeds.
	Jefferson County		
Sand	Sec. 25, T 3 S, R 5 W	20 m. W of Wheeler	Hillside quarry in ledge 25 ft. thick. Exposed 100 yd. Extensive deposit. Used for paving. Formerly extracted by water process.
Sand	NE ¼, Sec. 33, T 3 S, R 4 W	15 m. W of Wheeler	Continuation of previous deposit. Undeveloped.
	Stephens County		
Sand	SE ¼, Sec. 6, T 1 S, R 5 W	12 m. E of Duncan	Impregnated area along a fault. Undeveloped.
	Garvin County		
Sand	SE ¼, SW ¼, Sec. 36, T 1 N, R 3 W	2½ to 3 m. W of Hennepin	Hillside quarry. Exploration pit, undeveloped. Ledge 5 to 20 ft. thick.
	Carter County		
Sand	NW ¼, NE ¼, Sec. 26, T 5 N, R 1 E	5 m. SW of Ardmore	Large deposit about 30 yd. wide along fault. Exposed for about 440 yd. Extensively operated.
Sand	NW ¼, SE ¼, Sec. 14, T 5 S, R 1 E	8 m. SW of Ardmore	Large open quarry along fault. Outcrop 40 ft. by 300 ft. Vein traced about ¼ m. Contains 12–17% asphalt.
Sand	SE ¼, NW ¼, Sec. 10, T 5 S, R 1 E	6 m. SW of Ardmore	Hillside quarry in upturned area. Outcrop 20 by 50 yd.
Sand	SW ¼, NW ¼, Sec. 5, T 5 S, R 1 E	3 m. W of Waurika	Outcrop 12 by 45 ft., disappearing under soil. Undeveloped.
Sand	NE ¼, NW ¼, Sec. 21, T 4 S, R 12 E	6 m. NW of Ardmore	Quarry pit about 35 ft. thick and 40 ft. deep. Two veins separated by 5 ft. of shale. Extensive deposit. Formerly extracted.
Sand	SW ¼, Sec. 19, T 3 S, R 1 E	1 m. NW of Tiff	Quarry pit in Caddo Creek bottom. 25 ft. deep. Outcrop traced about ¼ m. Extensive deposit.
Sand	Sec. 12 and N ½ Sec. 13, T 3 S, R 2 W	18 m. NW of Ardmore	Large quarry pit about 100 ft. deep in vein 45 to 75 ft. wide. Vertical fault between shale. Water-extraction plant erected, but abandoned. Contains 12½% asphalt.

TABLE XVI

ROCK ASPHALTS IN OKLAHOMA—*Continued*

Base	Locality (S = Section, T = Township, R = Range)	Nearest City or Town	Remarks
	Carter County—Continued		
Sand	NW ¼, Sec. 11, T 2 S, R 2 W	25 NW of Ardmore	Hillside quarry in ledge 30 ft. thick. Exposed for 100 yd. Water-extraction plant erected, but abandoned.
Sand	SW ¼, NW ¼, Sec. 34, T 1 S, R 2 W	10 m. NW of Woodford	Hillside quarry in ledge 10 ft. thick. Outcrop about 200 yd. along stream. Overburden 4 to 8 ft. Undeveloped.
Sand	SW ¼, Sec. 9, T 1 S, R 2 W	10 m. NW of Woodford	Ledge 8 ft. thick along creek. Outcrops for 50 yd. Overburden 6 to 8 ft. thick. Undeveloped.
Sand	Center of E side Sec. 21, T 3 S, R 2 W	At Wheeler	Alternate layers of shale and asphaltic sand. Percentage of asphalt decreases with depth. In center of Wheeler oil field. Undeveloped.
Sand	NE corner Sec. 21, T 4 S, R 2 W	2 m. N of Hewett	Outcrop reported, but undeveloped.
	Murray County Buckhorn Region		
Sand and limestone	SE ¼, Sec. 14, T 1 S, R 3 E	9 m. NE of Dougherty	Mostly sand and trace of lime. Quarry pit in stratum 10 ft. thick. Only a moderate percentage of asphalt. Undeveloped.
Limestone	SE ¼, Sec. 23, T 1 S, R 3 E	½ m. NW of Buckhorn	Quarry pit in fine-grained limestone carrying 14 to 15% asphalt. Fossil shells in evidence. Vein 18 in. wide and 25 to 30 ft. long.
Sand and limestone	NE ¼, Sec. 28, T 1 S, R 3 E	7 m. NE of Dougherty	Outcrop. Undeveloped. Small and irregular percentage of asphalt. Underground mine, 180 ft. deep. Vein 18 ft. by 70 ft.
Limestone	S ½, SW ¼, NE ¼, Sec. 22, T 1 S, R 3 E	7½ m. NE of Dougherty	Contains 5–6% asphalt, also crystal calcite. Abandoned.
Sand	NE ¼, Sec. 28, T 1 S, R 3 E	7 m. NE of Dougherty	Quarry pit 75 ft. by 450 ft. Carries 6–8% asphalt. Abandoned.
Sand	SW ¼, SE ¼, Sec. 15, T 1 S, R 3 E	1 m. N of Buckhorn	Quarry pit in stratum 8 ft. thick, carrying 7–10% asphalt. Overburden 8–10 ft. thick of weathered asphaltic sandstone.
Sand	NW ¼, SE ¼, NW ¼, Sec. 22, T 1 S, R 3 E	6 m. NE of Dougherty	Pit in vein probably 25 ft. thick, carrying 7–8% asphalt. Undeveloped.
Sand and limestone	NW ¼, NW ¼, Sec. 22, T 1 S, R 3 E; also NE ¼, NE ¼, Sec. 21, T 1 S, R 3 E	6 m. NE of Dougherty	Three quarries, largest 100 ft. diam. in vein 8–10 ft. thick. Asphalt varies from a trace to 8–9%. Mottled appearance.
Limestone	SW ¼, SE ¼, Sec. 21, T 1 S, R 3 E	7 m. NE of Dougherty	Very large quarry in stratum 15–75 ft. thick, carrying 5–13% asphalt (Pen., at 77° F., 60–65). Rock granular to crystalline (calcite).
Sand and limestone	SE ¼, SE ¼, Sec. 21, T 1 S, R 3 E	7 m. NE of Dougherty	Continuation of previous vein. 6–12% asphalt (soft).
Limestone	NE ¼, SE ¼, Sec. 21, T 1 S, R 3 E	7 m. NE of Dougherty	Small pit in stratum 10 ft. thick. Varies in percentage of asphalt.

TABLE XVI

ROCK ASPHALTS IN OKLAHOMA—*Continued*

Base	Locality (S = Section, T = Township, R = Range)	Nearest City or Town	Remarks
	Murray County—Continued		
	Buckhorn Region—*Continued*		
Sand	SE ¼, SE ¼, Sec. 17, T 1 S, R 3 E	8 m. NE of Dougherty	Large quarry in vein 15 ft. thick, containing 10–12% asphalt. Overburden of conglomerate, 75 ft. thick. Outcrop 150 ft. (Volatile matter in extracted asphalt 325° F., 7 hrs., 6 per cent.)
Sand	NE ¼ Sec. 20, and NW ¼, Sec. 21, T 1 S, R 3 E	6 m. NE of Dougherty	Small undeveloped pit with asphalt seepage in evidence.
Sand	NW ¼, NE ¼, Sec. 29, T 1 S, R 3 E	6 m. NE of Dougherty	Large deposit in stratum 38 ft. thick, containing 7–8% asphalt. Overburden, of gravel 6–8 ft. thick.
	Brunswick Region		
Limestone	W ½ Sec. 25, and all Sec. 26, T 1 S, R 2 W	4 m. NE of Dougherty	Several quarries and pits in granular and crystalline rock, varying in percentage of asphalt (3–10%). Abandoned.
	Washita Canyon Region		
Limestone	Center of Sec. 21, T 1 S, R 2 E	4½ m. NE of Dougherty	Prospect outcrop. Variable percentage of asphalt. Undeveloped.
Limestone	Sec. 21, T 1 S, R 2 E	4 m. NW of Dougherty	Prospect of slightly impregnated rock in shales.
	Outlying Regions		
Sand	SW ¼ Sec. 20, and NE ¼ Sec. 29, T 1 N, R 4 E	5 m. NE of Sulphur	Outcrop containing 10–15% asphalt. Undeveloped
	Love County		
Sand	SW ¼ Sec. 6, T 6 S, R 2 E	¾ m. SE of Overbrook	Hillside quarry. Outcrop 40 yd. wide by 50 yd. long. Abandoned.
Sand	Sec. 27, T 6 S, R 2 E	N and S of Hickory Creek	Outcrop pits on top of hill. Undeveloped.
Sand	S½, Sec. 1, T 7 S, R 2 E	5 m. NE of Marietta	Oil springs emanating from asphaltic sand.
	Marshall County		
Sand	SW ¼, Sec. 32, T 4 S, R 5 E	2 m. W of Randolph	Low-grade asphaltic sandstone, associated with shale. Undeveloped.
Sand	SW ¼, Sec. 26, T 5 S, R 5 E	1½ m. NE of Madill	Outcrop 4 ft. thick. Exposed several hundred feet. Undeveloped.
Sand	NE ¼, Sec. 29, T 5 S, R 5 E	3 m. W of Madill	Sand asphalt lentil 8–10 ft. thick, carrying 4–5% asphalt.
Sand	NW ¼, Sec. 17, T 5 S, R 5 E	2 m. E of Oakland	Lentil 12 ft. thick, traced 400 yd. 1–5% asphalt. Undeveloped.
Sand	NW ¼, Sec. 26, T 7 S, R 5 E	18 m. S of Madill	Small lentils about 5 ft. thick in sandstone bluff. Undeveloped.

TABLE XVI

ROCK ASPHALTS IN OKLAHOMA—*Continued*

Base	Locality { S = Section, T = Township, R = Range }	Nearest City or Town	Remarks
	Johnston County		
Sand, limestone and shale	NW ¼, Sec. 6, T 4 S, R 5 E; also NE ¼ Sec. 1, T 4 S, R 4 E; also S ½, SE ¼. Sec. 36, T 3 S, R 4 E; also S ½, SW ¼, Sec. 31, T 3 S, R 5 E	3½ m. W of Ravia	Quarry 75 by 100 ft. in stratum 6 ft. thick, with overburden of shale. Contains 2½–13% asphalt (pen., 77° F., = 200), averaging about 7%. Crude material contains 65–75% soluble in HCl (limestone) and 15–20% mineral matter insoluble.
Sand	NE ¼, Sec. 14, T 4 S, R 5 E	1 m. N of Randolph	Stratum 6 ft. thick, carrying 4–5% asphalt. Undeveloped.
Sand	SE ¼, Sec. 19, T 4 S, R 6 E	5 m. N of Randolph	Open cut 20 by 50 ft. in stratum over 8 ft. thick. Undeveloped.
	Pontotoc County		
	Ada Region		
Sand	NW ¼, NW ¼, Sec. 31, T 4 N, R 6 E	3 m. W of Ada	Outcrop traced several miles along Sandy Creek, with numerous seepages of asphalt. Many quarries and pits.
Sand	NE ¼, Sec. 5, T 2 N, R 5 E	2 m. W of Fitzhugh	Asphalt sand with seepages of oil and asphalt. Undeveloped.
	Roff Region		
Sand	Sec. 15, T 2 N, R 4 E	2 m. NW of Roff	Quarry in strata 10 ft. thick. Abandoned.
Sand, limestone and shale	Sec. 28, and Sec. 29, T 2 N, R 6 E	9 m. E of Roff	Several large quarries in thick stratum. Abandoned.
	Atoka County		
Shale	Center Sec. 35, T 2 S, R 13 E	18 m. SE of Atoka	Several prospect pits 10–15 ft. deep. Small amount of asphalt.
Sand	NE ¼, Sec. 13, T 1 S, R 14 E	15 m. W of Frisco R. R.	Ledge of unknown thickness, carrying 6–8% asphalt.
	McCurtain County		
Sand	Sec. 20, T 7 S, R 24 E	3 m. NE of Ada	Ledge 25 ft. thick, running ½ m. along stream. Very large deposit containing a large percentage of asphalt. Undeveloped.
	Leflore County		
Sand	Sec. 23, T 3 N, R 26 E	1 m. E of Page	Outcrop carrying 6% asphalt. Undeveloped.

The deposits consist of asphaltic sands, asphaltic limestone, mixtures of the two, and rarely asphalt impregnated shale. The principal occurrences are included in Table XVI.[5]

In the majority of cases the asphaltic impregnation is of liquid to semi-liquid consistency, having a comparatively low fusing-point. It is contended by some authorities that the vast deposits of sand asphalt previously constituted oil-sands which have been laid bare by the agencies of erosion, faulting, crumpling and upturning of the

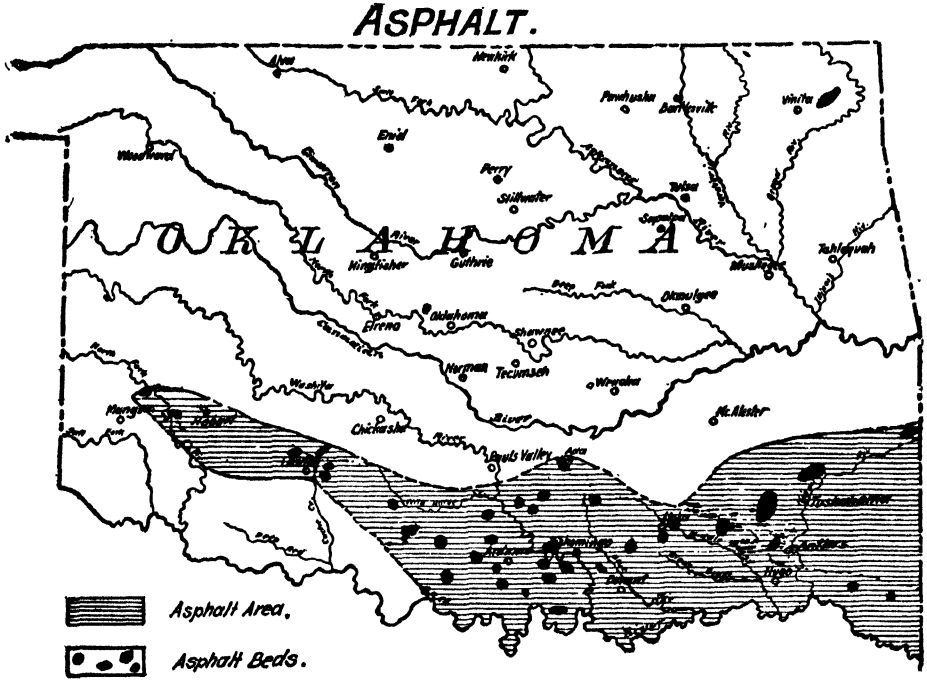


FIG. 48.—Map of Asphalt Region in Oklahoma.

strata, so that the lighter oils and gases have escaped into the air, leaving the sand impregnated with the comparatively non-volatile asphaltic constituents. Most of the deposits occur along pronounced fault lines, although faulting is not essential, since certain deposits have become impregnated by the uprising of asphalt-bearing petroleum from regions below, through the porous sandstone or limestone.

A characteristic feature of these deposits is the sand grains, which are round and unconsolidated, being held together by the asphalt filling the voids. When the asphalt is extracted the grains

fall apart, and show the same general characteristics as an ordinary petroleum-bearing sand.

The extent of these deposits has been variously estimated from 2 to 13 million tons.[6]

Most of the asphalt mined in Oklahoma has been used for paving purposes, and the author has seen many satisfactory pavements laid through the state which have withstood the wear and tear of traffic, also exposure to the elements. It is generally necessary to modify the rock asphalts either by combining the products obtained from different deposits, or by incorporating pure sand, until a proper balance is obtained between the asphalt and the mineral constituents. In general, the best results have been obtained with mixtures containing 7 to 10 per cent of asphalt in the finished paving composition.

Numerous water-extraction plants have been erected to separate the asphalt from the sand, but most of these have proven unsuccessful, since the extraction process raises the price of the refined asphalt so that it is unable to compete with petroleum asphalts obtained from other sources in the neighborhood.

Tests made with sand asphalt taken from the quarry in Carter County, Sec. 12 and N ½ Sec. 13, T 3 S, R 2 W, 18 miles northwest of Ardmore, indicated the following. The dry sand asphalt contained 12.5 per cent of pure asphalt having a fusing-point (K. and S. method) between 65 and 69° F. On subjecting it to the water-extraction process, the following results were recorded:

	Products Recovered, Per Cent.	Asphalt Content, Per Cent.	Total Pure Asphalt, Per Cent.
Asphalt recovered	6	95	5.7
Impure asphaltic residue	3	60	1.8
Separated sand waste	91	2½	2.3
Volatile asphalt	..	..	2.7
Asphalt in crude rock	..	..	12.5
Total	100	..	..

On boiling the crude rock with water, impure asphalt rises to the surface, and the "sand waste" settles to the bottom. Upon dehy-

drating the impure asphalt, more sand settles out, constituting what is designated "impure asphaltic residue." The pure asphalt drawn off from this residue is termed "asphalt recovered."

The "asphalt recovered" contained 5 per cent of mineral matter and tested as follows:

(Test 9*b*)	Penetration at 32° F.	85
	Penetration at 77° F.	Over 360
	Penetration at 115° F.	Too soft
(Test 9*c*)	Consistency at 32° F.	10.0
	Consistency at 77° F.	1.5
	Consistency at 115° F.	0.0
(Test 9*d*)	Susceptibility index	15
(Test 15*a*)	Fusing-point (K. and S. method)	65–69° F.
(Test 15*b*)	Fusing-point (R. and B. method)	78–87° F.
(Test 16)	Volatile at 500° F. in 5 hrs.	10 per cent

Examination of Residue (from Test 16)

(Test 9*b*)	Penetration at 32° F.	7
	Penetration at 77° F.	58
	Penetration at 115° F.	250
(Test 9*c*)	Consistency at 32° F.	72.6
	Consistency at 77° F.	10.7
	Consistency at 115° F.	1.1
(Test 9*d*)	Susceptibility index	63.1
(Test 10*b*)	Ductility at 77° F.	Over 100
(Test 15*a*)	Fusing-point (K. and S. method)	115° F.
(Test 15*b*)	Fusing-point (R. and B. method)	131° F.

Upon evaporating the "asphalt recovered" at 250–260° C., the following figures were recorded:

Total Loss, Per Cent.	Fusing-point °F. (Test 15*a*)	Penetration at 77° F. (Test 9*b*)
15	120	40
20	125	19
25	147	10
27	165	4½

A sample of the "asphalt recovered" upon being blown with dry air at 300° C. for nine hours, lost 23 per cent in weight, showed a fusing-point of 165° F., and a hardness at 77° F. of 38.0 (Test 9*c*), corresponding to a penetration at 77° F. of 8 (Test 9*b*). It is apparent that the extracted asphalt is scarcely affected by blowing, and thus differs from asphalts obtained upon distilling petroleum. It

has been reported that blowing decreases the percentage of asphaltous acids, anhydrides, oily constituents and resins. The molecular weight of the resins increases from 733 to 1012 and that of the asphaltenes from 2219 to 4690 during the blowing process.[7] It has been further corroborated by the author's observations on paints made from the extracted sand asphalt, which were found to be *highly resistant* to atmospheric oxidation. A sample spread on cloth and exposed to air indoors for about a year, showed scarcely any diminution in tackiness.[8] Petroleum asphalts of the same consistency when tested in a similar manner, dry out in a much shorter time.

A mixture containing 82 per cent of the "asphalt recovered" fluxed with 18 per cent of grahamite, showing the same fusing-point (165° F.), tested as follows:

(Test 7)	Specific gravity at 77° F.	1.09
(Test 9*b*)	Penetration at 115° F.	26
	Penetration at 77° F.	17
	Penetration at 32° F.	9
(Test 9*c*)	Consistency at 115° F.	14.7
	Consistency at 77° F.	27.1
	Consistency at 32° F.	65.4
(Test 9*d*)	Susceptibility index	30.7
(Test 10*b*)	Ductility at 115° F.	4.5
	Ductility at 77° F.	1.0
	Ductility at 32° F.	0.0
(Test 11)	Tensile strength at 115° F.	1.8
	Tensile strength at 77° F.	6.5
	Tensile strength at 32° F.	9.5
(Test 15*a*)	Fusing-point (K. and S. method)	165° F.
(Test 15*b*)	Fusing-point (R. and B. method)	183° F.
(Test 16)	Volatile at 500° F. in 5 hrs.	0.5 per cent

Arkansas.

Seven to eight known deposits of asphalt occur in southwestern Arkansas, in Pike and Sevier Counties, only one of which has been developed into a mine producing commercial quantities. This is located 2½ miles south-southeast of Pike, in Pike County.[9] The asphalt impregnates nearly horizontal strata of unconsolidated sand, except in one locality, where it impregnates gravel. The veins run from 1 in. to 12 ft. thick.

Pike County. The most important deposit occurs on the west side of the road connecting the towns of Pike and Delight, about 2½ miles south-southeast of Pike. It has been mined in an open

cut 100 ft. wide, 200 ft. long and 15 ft. deep. The sand carries from 5 to 16 per cent asphalt, averaging 7½ per cent, and is used for paving purposes. Prospects occur about 4 miles north-northwest of Delight in a bed 3 to 5 ft. thick, carrying up to 17 per cent asphalt associated with sand carrying a few nodules of iron pyrites; also about 1 mile northeast of Murfreesboro, on the east bank of Prairie Creek, in which the asphalt impregnates gravel.

Sevier County. Prospects occur ½ mile southeast of Lebanon, alongside of the road adjoining the Salina River bottom, in lenticular formations up to 1 ft. thick; another about 2½ miles west-southwest of Lebanon; another on the south side of the road 4 miles west-southwest of Lebanon; and still another 6½ miles west-southwest of Lebanon along the road just northwest of Moody Shoal Ford on Cossatot River.

Alabama.

An area occurs in northern Alabama measuring about 72 miles by 8 miles in which 1 to 15 per cent of asphalt is associated with unconsolidated sand granules. It extends through the following counties:[10]

Colbert County. A fairly extensive deposit of sand asphalt has recently been developed near Florence, on the banks of the Tennessee River, in the vicinity of Muscle Shoals, which has been used in the vicinity for paving purposes. Another deposit occurs 1 mile south of Margerum, on the Southern Railroad, within an area 3 miles square, consisting of asphaltic limestone. Others have been reported 2 miles southeast of Cherokee, consisting of asphaltic limestone and sand; likewise sand asphalt deposits 1 to 7 miles east of Littleville, and at Leighton and Russelville (extending into Franklin County).

Lawrence County. Asphalt sand is found at Wolf Springs on the summit of Little Mountain, about 6 miles southwest of Town Creek Station on the Southern Railroad. Another deposit occurs at Caddo, midway between Moulton and the Morgan County line, in the southeast section.

Morgan County. A sand asphalt deposit occurs about 3 miles northwest of Flint Station on the L. & N. Railroad, and another in the vicinity of Hartsville, to the east and southeast thereof.

Louisiana.

Lafayette Parish. A sand asphalt deposit has recently attracted attention about 5 miles from Lafayette, covering about 50 acres on the surface.[11]

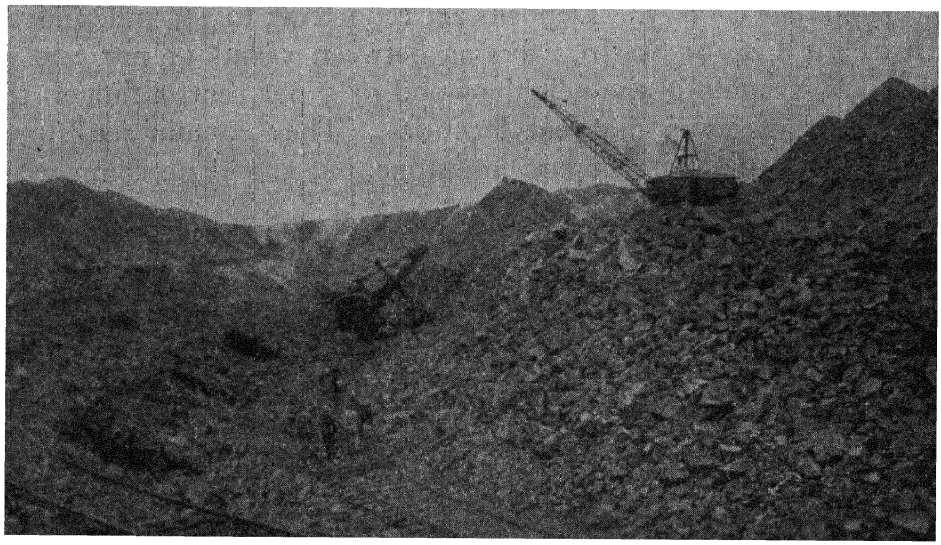
Texas.[12]

Montague County. Deposits are reported 3 to 3½ miles northeast of the City of St. Jo, carrying between 5 and 11 per cent of asphalt, averaging in the neighborhood of 7 per cent, although the percentage varies in different localities. They contain sandstone, or a mixture of sandstone and limestone, but are of no commercial importance.

Burnet County. This occurrence is at Post Mountain near the town of Burnet, and consists of an asphaltic limestone, containing about 10 per cent of asphalt of a very soft consistency (having a penetration of 20–250 at 77° F.).

Uvalde County. The most important Texas deposits are found in the southwestern part of this county near Cline, about 18 to 25 miles west of the city of Uvalde, in the region of the Anacacho Mountains. They consist of limestone, carrying 10 to 20 per cent of asphalt, averaging about 15 per cent. Crystalline calcite is present, also numerous fossil remains of molluscs, indicating the asphalt to be of animal origin. The deposits have been traced for several miles, but their exact extent is not accurately known. A large quantity has been quarried, and from recent reports the mines are still being operated. The impregnating asphalt is quite hard, showing a conchoidal fracture and brilliant lustre. It has a moderately high fusing-point, and analyzes: carbon 81 per cent, hydrogen 12 per cent, sulfur 6½ per cent, nitrogen ½ per cent; total 100 per cent. The rock asphalt is too hard for a paving material if used alone. It is therefore mixed with about 25 per cent of trap rock and the requisite amount of flux. The mixture is adapted to be rolled *cold* in surfacing a pavement. One ton will cover 20 sq. yd. 1 in. thick. Fig. 49 illustrates the principal mine, with about 80,000 tons of asphalt rock blasted loose and ready to be loaded into the cars by the steam shovel. It is then transported to the mixing and crushing plant. Fig. 50*A* shows the equipment for crushing and screening the rock asphalt; Fig. 50*B* the device for mixing the trap

rock with the rock asphalt; and Fig. 50*C* shows a 42 cu. ft. capacity twin pug-mill which is used for pulverizing the asphalt. The pure asphalt extracted from the rock has been marketed under the name "litho-carbon" [13] which may also be blown. This constitutes the most important deposit in Texas.



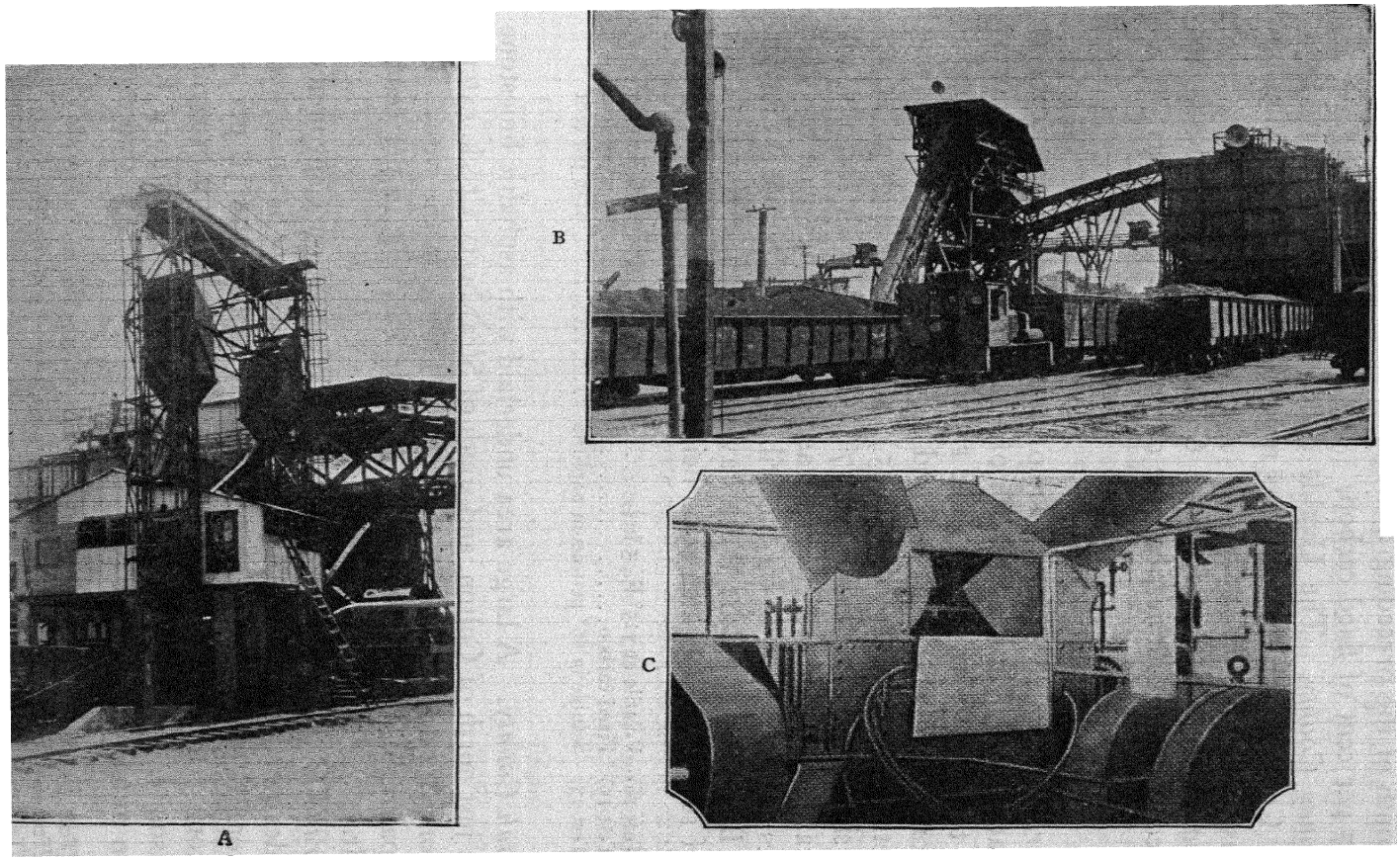
Courtesy Uvalde Asphalt Company.

FIG. 49.—Rosk-asphalt Quarry in Uvalde County, Texas.

Litho-carbon shows the following tests:

(Test 1)	Color in mass	Black
(Test 2)	Fracture	Conchoidal
(Test 3)	Lustre	Bright
(Test 4)	Streak	Brownish black
(Test 7)	Specific gravity at 77° F.	1.119
(Test 9*b*)	Penetration at 115° F.	16
	Penetration at 77° F.	4
(Test 15*a*)	Fusing-point (K. and S. method)	116° F.
(Test 16)	Volatile at 500° F., in 5 hours	2.76 per cent
(Test 21)	Soluble in carbon disulfide	96.35 per cent
	Non-mineral matter insoluble	0.75 per cent
	Mineral matter	2.90 per cent
	Total	100.00 per cent
(Test 23)	Soluble in 88° petroleum naphtha	53.08 per cent

Other deposits of the same general character are found in the neighborhood. One 20 miles south-southwest of Uvalde and 5



Courtesy Uvalde Asphalt Company.

FIG. 50.—Uvalde Rock-asphalt Plant. *A* represents the equipment for crushing and screening mine-run asphalt, *B* represents the mixing plant, and *C* the pulverizing mill.

miles south of the preceding quarry showed 12 per cent of asphalt with 17 per cent of fixed carbon.

Kinney County. The Uvalde deposits extend westward into Kinney County, with outcroppings near Blewett, where mining operations have been undertaken.

Anderson, Jasper, and Cooke Counties. Minor deposits are reported in these counties, but are of no commercial value.[14]

Utah.

Carbon County.[15] A deposit of asphaltic limestone occurs at the head of the right-hand branch of Pie Fork, a canyon northwest of the town of Clear Creek. The rock is non-uniform in composition, some containing between 6 and 14 per cent of asphalt (having a penetration at 77° F. of 7 to 15), and some as high as 75 per cent (showing a penetration at 77° F. of 45) with scarcely any fixed carbon. Another deposit of asphaltic sand has been reported 8 miles from Sunnyside on the tributaries of Whitmore Canyon, in the Brook Cliffs on the West Tavaputs Plateau, carrying 11 per cent of soft asphalt, which upon being extracted tested as follows:

(Test 16)	Volatile at 325° F., 5 hrs	6.6 per cent
(Test 19)	Fixed carbon	5.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	91.8 per cent

Utah County. A large area underlaid with asphaltic limestone occurs just north of Colton, and south of Strawberry Creek, extending from Antelope Creek on the east to Thistle on the west. The principal deposit is at the town of Asphalt, carrying 12 per cent of asphalt.

Grand County. At the head of the West Water Canyon about 20 miles north of the town of West Water, there is an asphaltic limestone deposit containing 50 per cent asphalt and 50 per cent limestone. Investigations indicate that this asphalt is a progenitor of gilsonite. The extracted asphalt is reported by Clifford Richardson to test as follows:

(Test 7)	Specific gravity at 77° F.	1.037
(Test 9*b*)	Penetration at 77° F.	22
(Test 16)	Volatile at 212° F.	2.8 per cent
(Test 19)	Fixed carbon	8.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	88.7 per cent

Uinta County. The largest deposit of asphaltic sandstone occurs 3 to 4 miles southwest of Vernal, north of the Green River, between the Ashley and Uinta Valleys, in an outcrop about 11½ miles long, known as "Asphalt Ridge." [16] It contains 8 to 15 per cent by weight of asphalt, averaging 11½ per cent. A typical specimen analyzed as follows:

(Test 21)	Soluble in carbon disulfide	12.8 per cent
	Mineral matter on ignition	86.45 per cent
(Test 16)	Volatile at 105° C. in 1 hour	0.20 per cent

The extracted asphalt tested as follows:

(Test 7)	Specific gravity at 77° F.	0.980
(Test 19)	Fixed carbon	7.1 per cent

The asphalt has been used successfully just as it is mined, for paving the streets of Vernal.

Another deposit, or rather a series of deposits, occur in Argyle Creek, a tributary of the Minnie Maud Creek, which in turn flows into the Green River about 20 miles south of Ouray. The material consists of an asphaltic sandstone, exploited under the name "argulite," carrying between 8 and 10 per cent of asphalt. The extracted asphalt tests as follows:

(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	0.997–1.013
(Test 9*b*)	Penetration at 77° F.	14
(Test 16)	Volatile at 325° F. in 5 hrs.	25.8 per cent
(Test 19)	Fixed carbon	8.55
(Test 23)	Soluble in 88° petroleum naphtha	88
(Test 26)	Carbon	89.9 per cent
(Test 27)	Hydrogen	9.0 per cent
(Test 28)	Sulfur	0.0 per cent
(Tests 29 and 30)	Nitrogen and oxygen	1.1 per cent
	Total	100.0 per cent
(Test 34*a*)	Saturated hydrocarbons	25 per cent

Wyoming.

Fremont County. Asphalt and semi-solid asphalt seepages were used for many years as fuel by shepherds on Copper Mountain in section T. 4 N., R. 92–93 W., in northeastern Fremont County.

California.[17]

Mendocino County. Deposits of asphaltic sand are found 2 miles north of the town of Point Arena and ½ mile from the coast, carrying between 6 and 7 per cent of asphalt. A similar deposit occurs just north of Port Gulch.

Santa Cruz County. Large deposits of asphalt sand occur 4 to

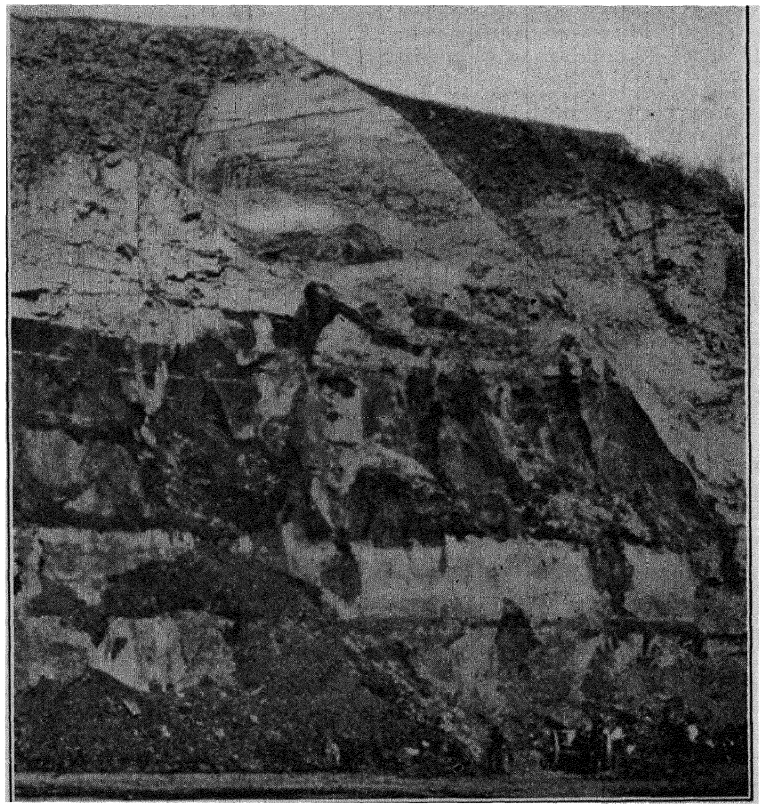


FIG. 51.—Sand Asphalt Quarries in Santa Cruz County, Cal.

6 miles northwest of the city of Santa Cruz, near the summit of Empire Ridge, a spur of the Santa Cruz Mountains, 3½ miles from the coast. A number of quarries have been opened up in this region, and the product used for constructing pavements in Santa Cruz and San Francisco. The rock contains between 10 and 17½ per cent asphalt of variable hardness, containing a substantial proportion of volatile matter. The veins vary from 2 to 32 ft. thick,

as shown in Figs. 51 and 52. Three strata are found, an upper 8 ft. thick containing 2–3 per cent asphalt, a middle one 32 ft. thick containing 14 per cent asphalt, and a lower 25 ft. thick carrying 16–18 per cent asphalt.

Monterey County. Several deposits of asphaltic sandstone are scattered throughout the Salinas Valley. A prospect occurs about 10 miles from King City, composed of particles of quartz, feldspar and mica, impregnated with a varying percentage of asphalt. Another deposit occurs 7 miles southeast of Metz at the head of

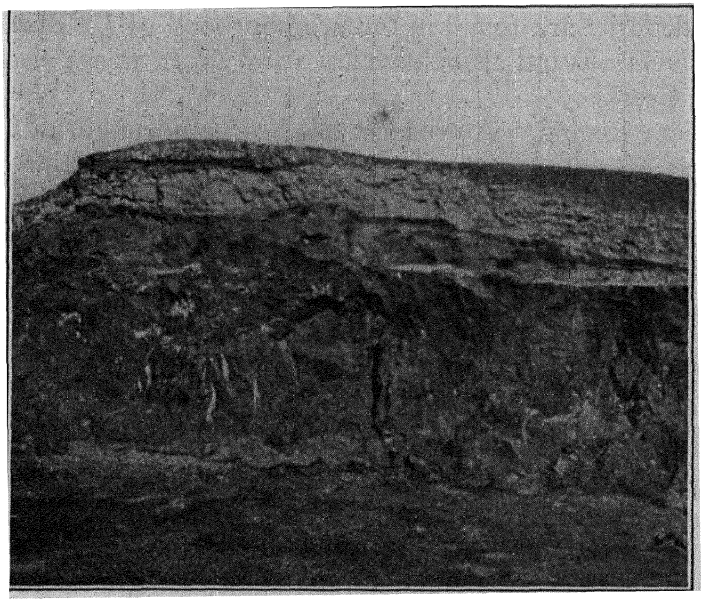


FIG. 52.—Sand Asphalt Quarries in Santa Cruz County, Cal.

Chelone Creek, of the same general character. A large vein, about 125 ft. thick and 3 miles long, has been reported near San Ardo, composed of coarse quartz grains, and a little feldspar, impregnated with a small percentage of asphalt.

San Luis Obispo County. Sand asphalt deposits occur about 80 miles southwest of the town of San Luis Obispo, consisting of a number of actively worked quarries. The rock is fine grained, of even texture, consisting mostly of quartz, with a small quantity of feldspar. The percentage of asphalt varies from 8 to 18 per cent, averaging about 10.

Santa Barbara County. Santa Maria Region. Associated with the pure asphalt deposits already described, zones of asphalt-impregnated shale have been reported on the western slope of the Azufre Hills, containing 30 to 40 per cent of asphalt.

Sisquoc Region. Deposits of sand asphalt occur in the neighborhood of the town of Sisquoc, carrying between 14 and 18 per cent of asphalt. The largest vein occurs in Bishop's Gulch, about 100 ft. thick, running fairly uniform in composition. Some time ago an attempt was made to remove the asphalt by extraction with solvents, but the process proved too costly and had to be abandoned. Similar deposits are found in the neighborhood of La Brea Creek, where a vein of sand asphalt occurs 20 to 60 ft. thick; also at Los Alamos Creek.

Gaviota Region. A prospect has been reported in this locality consisting of a bed of sandstone and conglomerate about 25 ft. thick, containing 7 to 8 per cent of asphalt.

Mores' Landing. This deposit is found on the seacoast about 7 miles west of Santa Barbara, occurring as veins and irregular masses in massive sandstone cliffs, at least 100 ft. thick. According to Eldridge it contains 30 to 60 per cent of asphalt and has a strong resemblance in structure, brilliancy, and fracture to gilsonite, although it is very much softer in consistency.

La Patera Region. A vein of asphalt of historical interest only, occurs about 10 miles west of Santa Barbara, close to the coast. It varies in width from 2 to 12 ft., with a number of lateral branches several inches thick. The asphalt is associated with 30 to 50 per cent of mineral matters composed of shale, sand, and clay. It is stated that 30,000 tons have been removed from this mine, testing, when dried, as follows: [18]

(Test 4)	Fracture	Irregular
(Test 5)	Lustre	Dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.38
(Test 9*a*)	Hardness on Moh's scale	2
(Test 9*b*)	Penetration at 77° F	0
(Test 16)	Volatile at 400° F., 5 hrs	2.5 per cent
(Test 19)	Fixed carbon	14.9 per cent
(Test 21)	Soluble in carbon disulfide	Approx. 50 per cent
	Mineral matter	About 50 per cent
(Test 23)	Soluble in 88° petroleum naphtha	21.6 per cent
(Test 28)	Sulfur	6.2 per cent
(Test 34*a*)	Saturated hydrocarbons	8.1 per cent

Carpinteria Region. This deposit, composed of asphaltic sand about 15 ft. thick, lying along the ocean's shore at Benham, 2 miles southeast of Carpinteria, is illustrated in Fig. 53. It contains 18 to 20 per cent of asphalt filling the interstices of unconsolidated quartz grains. Some time ago a process was installed for extracting the asphalt with water, but this never proved successful commercially.

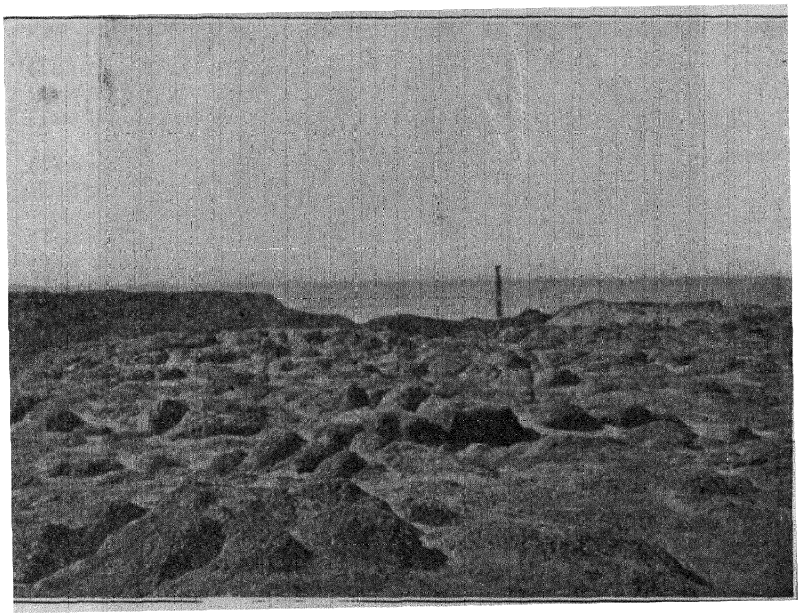


FIG. 53.—Asphaltic Sand on the Shore at Carpinteria, Cal.

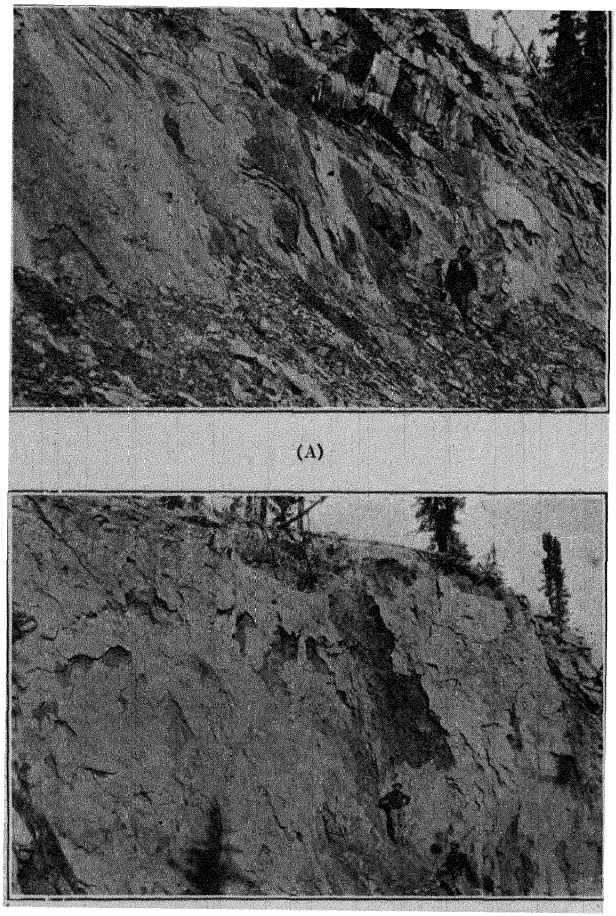
Orange County. Bituminous sands have been reported 4 miles southwest of Chino, in a layer about 6 ft. thick, containing varying percentages of asphalt.

CANADA

Alberta Province.

McMurray Region. Vast deposits of asphaltic sands occur on both banks of the Athabaska River, and its tributary, the Clear Water River, covering probably not less than 750 square miles.[19] The deposit varies in thickness to a maximum of 225 ft. Characteristic views of the outcrop on the Athabaska River are shown in Fig. 54 (A and B). The material contains 12 to 20 per cent as-

phalt, averaging between 15 and 18 per cent, associated with an unconsolidated sand consisting of between 93 and 99 per cent pure



(A)

(B)

Courtesy S. C. Ells.

FIG. 54.—Asphaltic Sand on Banks of Athabaska River, Alberta, Can.

silica. A marked variation in the size of the sand grains is characteristic of almost every exposed section of the deposit, ranging from 40 to 80 mesh. The asphalt is amenable to the water extraction

process. A specimen of the extracted asphalt examined by the author tested as follows:

(Test 7)	Specific gravity at 77° F.	1.022
(Test 9*b*)	Penetration at 77° F.	Too soft for test
	Penetration at 32° F.	120
(Test 9*c*)	Consistometer hardness at 115° F.	0.0
	Consistometer hardness at 77° F.	0.0
	Consistometer hardness at 32° F.	2.7
(Test 10*b*)	Ductility at 115° F.	2.0
	Ductility at 77° F.	7.0
	Ductility at 32° F.	12.5
(Test 15*a*)	Fusing-point (K. and S. method)	50° F.
(Test 15*b*)	Fusing-point (R. and B. method)	63° F.
(Test 16)	Volatile at 500° F. in 5 hrs.	17.9 per cent
	Volatile at 325° F. in 5 hrs.	11.2 per cent
(Test 19)	Fixed carbon	7.23–10.55 per cent
(Test 21)	Soluble in carbon disulfide	97.3 per cent
	Mineral matter	2.7 per cent
(Test 23)	Soluble in 88° petroleum naphtha	78.2 per cent
(Test 26)	Carbon	84.49 per cent
(Test 27)	Hydrogen	11.23 per cent
(Test 28)	Sulfur	2.73 per cent
(Test 29)	Nitrogen	0.04 per cent
	Total	98.49 per cent
(Test 34*a*)	Saturated hydrocarbons	39.6 per cent

The non-volatile matter tested as follows:

		Residue at 500° F.	Residue at 325° F.
(Test 7)	Specific gravity at 77° F.	1.028	1.021
(Test 9*b*)	Penetration at 115° F.	110	.....
	Penetration at 77° F.	62	262
	Penetration at 32° F.	18	.....
(Test 9*c*)	Consistency at 115° F.	3.7	.....
	Consistency at 77° F.	8.5	.....
	Consistency at 32° F.	49.3	.....
(Test 9*d*)	Susceptibility index	36.5	.....
(Test 10*b*)	Ductility at 115° F.	34.5	+100
	Ductility at 77° F.	45.0	+100
	Ductility at 32° F.	0.5	.....
(Test 11)	Tensile strength at 115° F.	0.3	.....
	Tensile strength at 77° F.	1.5	.....
	Tensile strength at 32° F.	25.5	.....
(Test 15*a*)	Fusing-point (K. and S. method).	125° F.	106° F.
(Test 15*b*)	Fusing-point (R. and B. method)	142° F.	121° F.
(Test 19)	Fixed carbon	12.33 per cent	8.99 per cent

A sample of extracted Alberta asphalt having a K. and S. fusing-point of 84° F. was blown 2½ hours at 500° F. during which

process it lost 17 per cent in weight, and the resultant product tested as follows:

(Test 5)	Lustre	Bright and tough
(Test 9*b*)	Penetration at 115° F	5.6
	Penetration at 77° F	3.0
	Penetration at 32° F	1.0
(Test 15*a*)	Fusing-point (K. and S. method)	208° F.

A duplicate sample was evaporated in air until it lost exactly 17 per cent by weight, whereupon the residue showed a fusing-point of 128° F. (K. and S.), demonstrating that the material was susceptible to the process of blowing.

The crude asphalt, after being tempered with additional pure sand to reduce the percentage of asphalt, has given successful results for paving purposes in Edmonton, Canada.

Manitoba Province. Similar deposits are reported to occur in the Clear River District in this province.[20]

MEXICO

States of Vera Cruz and Tamaulipas. Several deposits of sand asphalt have been reported in the neighborhood of Tampico and Vera Cruz, containing 8 to 14 per cent of asphalt, but none have been developed commercially.

CUBA[21]

Province of Matanzas. Semi-solid asphalts have been mined for many years at the bottom of Cardenas Harbor.[22] The most important deposit, known as the "Constancia Mine," occurs about 12 ft. below the level of the water, and is consequently mined with difficulty. Other deposits of semi-liquid asphalt containing more or less mineral matter, occur at the mouth of the La Palma River, about 20 miles from Cardenas; also near Sabanilla de la Palma, about 30 miles east of Cardenas and 4 to 5 miles west of Hato Nuevo. Analyses are not available.

Province of Pinar del Rio. Deposits of sand asphalt have been reported at Bahia Honda and Mariel, in the neighborhood of Mariel Bay, also at Vuelta Abajo. No analyses are available.

Province of Havana. An extensive deposit approaching glance

pitch in properties, known as the "Angelo Elmira Mine," has been found near Bejucal, about 18 miles south of Havana, associated with mineral matter composed of calcium carbonate, silica, and silicates, which, according to Clifford Richardson,[23] tests as follows:

(Test 1)	Color in mass	Brownish black to black
(Test 4)	Fracture	Semi-conchoidal to conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Reddish brown to brown
(Test 7)	Specific gravity at 77° F.	1.30–1.35
(Test 9*a*)	Hardness, Moh's scale	2–3
(Test 9*b*)	Hardness, penetrometer at 77° F.	0
(Test 16)	Volatile at 325° F., 5 hrs. (dry substance)	About 1 per cent
	Volatile at 400° F., 5 hrs. (dry substance)	About 1½ per cent
(Test 19)	Fixed carbon	17.4–25.0 per cent
(Test 21)	Soluble in carbon disulfide	70–75 per cent
	Non-mineral matter insoluble	3½– 4 per cent
	Mineral matter (calcium carbonate, etc.)	21–28 per cent
(Test 23)	Soluble in 88° petroleum naphtha	32–50 per cent
(Test 28)	Sulfur	About 8.3 per cent

Five miles east of Bejucal, there is another deposit, similar to the preceding.

Province of Camaguey. Impure soft and hard asphalt deposits are found near Minas, a small town about 30 miles from Nuevitas, and 20 miles from Puerto Principe.

Province of Santiago de Cuba. A deposit of hard impure asphalt occurs 5 to 10 miles south of Puerto Padre in Victoria de las Tunas district, testing as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Hackly
(Test 5)	Lustre	Dull
(Test 6)	Streak	Dark brown
(Test 7)	Specific gravity at 77° F.	1.106
(Test 9*a*)	Hardness, Moh's scale	1
(Test 19)	Fixed carbon	9.9 per cent
(Test 21)	Soluble in carbon disulfide	78.4 per cent
	Non-mineral matter insoluble	18.2 per cent
	Mineral matter	3.4 per cent
	Total	100.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	60.6 per cent

SOUTH AMERICA

TRINIDAD

St. Patrick County. One of the largest deposits of asphalt in the entire world occurs on the Island of Trinidad[24] on the north coast of South America, situated a short distance from the mainland of Venezuela, between the Caribbean Sea on the west and the Atlantic Ocean on the east.

Small deposits are scattered all over the Island, but the largest one known as the "Trinidad Asphalt Lake," is situated on La Brea Point, in the Wards of La Brea and Guapo, on the western shore. The lake is situated on the highest part of La Brea Point, 138 ft. above sea level. It covers an area nearly circular comprising 115 acres, in a slight depression or shallow crater at the crest of the hill. The exact location of the lake is shown in Fig. 55. The lake measures about 2000 ft. across and is over 135 ft. deep in the center, becoming shallower towards the edges. A panoramic view is shown in Fig. 56.

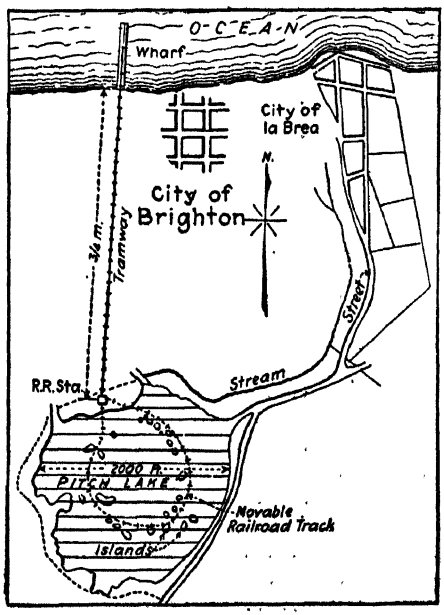
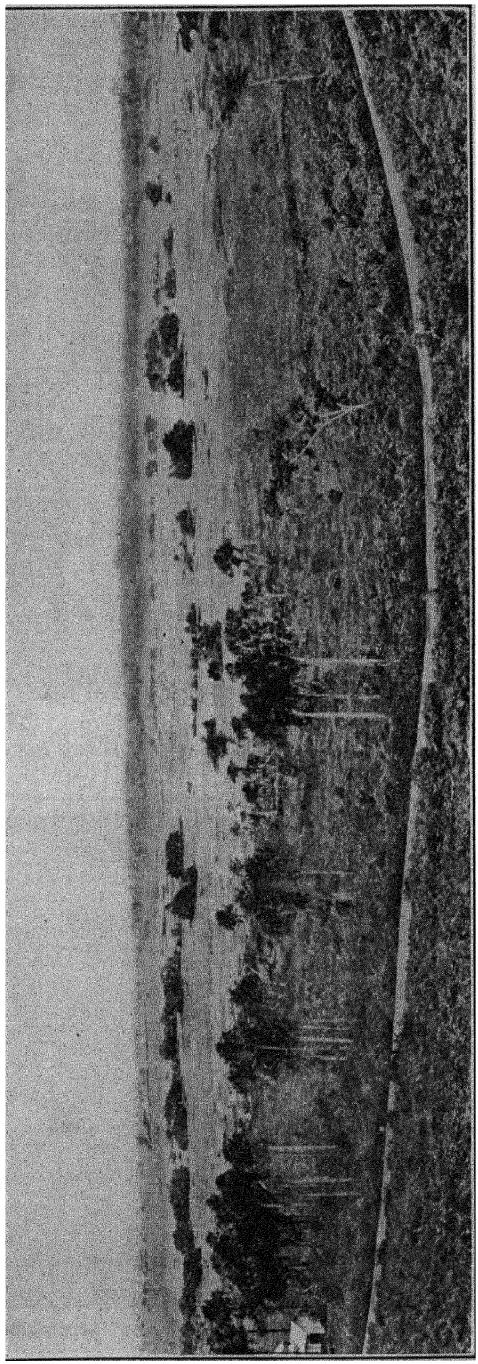


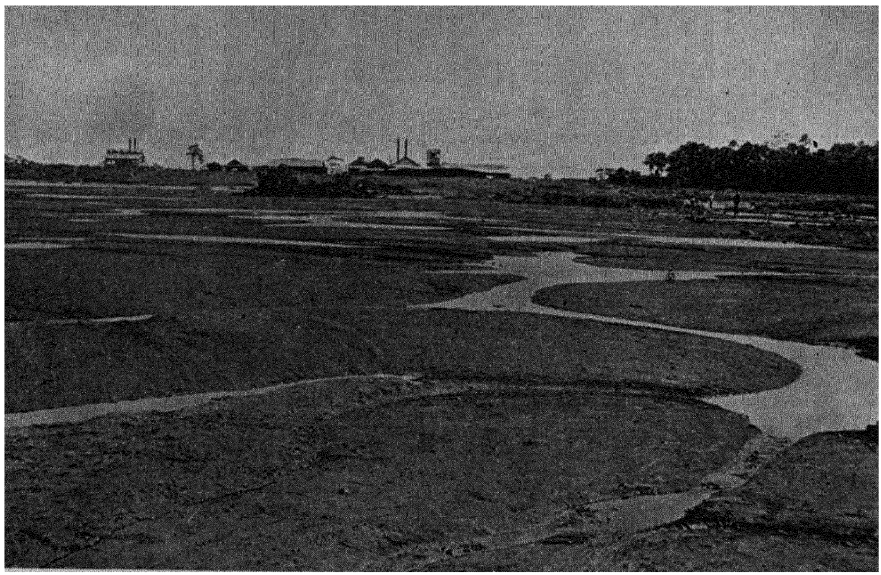
FIG. 55.—Map of Trinidad Asphalt Lake.

The asphalt surface is broken up into a series of large folds with accumulations of rain water in the creases. A typical view is shown in Fig. 57. The entire mass of asphalt is in constant but slow motion from the center towards the edges, probably due to the continual influx of solid material at the center, accompanied by a strong evolution of gas which imparts a porous or honeycombed structure. The evolution of gas through the water is shown in Fig. 58. Wherever a hole is dug in the surface, it slowly fills up and disappears. The asphalt is softest in the center of the deposit, and gradually hardens towards the circumference. Even in the center, the consistency is such that it



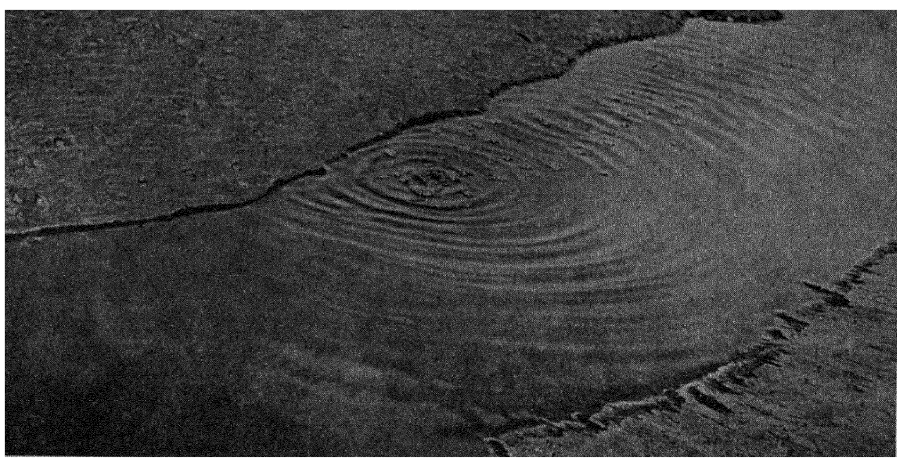
Courtesy of Barber Co.

FIG. 56.—Panoramic View of Trinidad Lake.



Courtesy of Barber Co.

FIG. 57.—Folds in the Surface of Trinidad Lake.



Courtesy of Barber Co.

FIG. 58.—Evolution of Gas from Trinidad Lake.

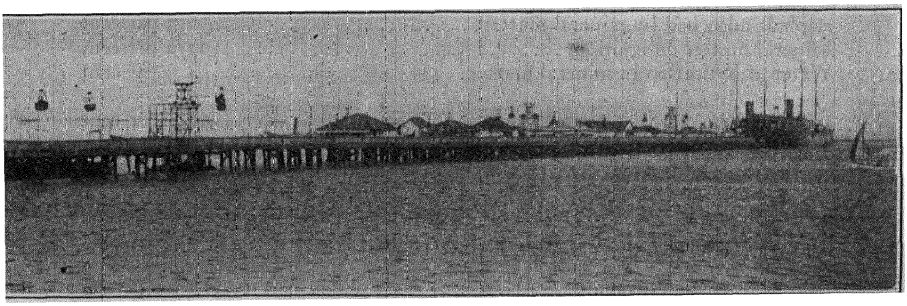
will bear the weight of a man, and can be readily broken out in large masses with picks as shown in Fig. 59.

Shrubs and small trees grow on the surface in isolated patches known as "islands," which slowly migrate from place to place with



Courtesy of Barber Co.

FIG. 59.—Gathering Trinidad Lake Asphalt.



Courtesy of Barber Co.

FIG. 60.—Transporting Trinidad Lake Asphalt.

the movement of the asphalt. Grassy vegetation extends along the edges of the lake merging into the surrounding country.

The crude asphalt is loaded on small cars run by cable over the lake in a loop, the rails being supported by wooden ties which must be replaced from time to time as they gradually sink into the surface of the asphalt. The asphalt is transferred to an inclined cable way

at the end of the loop which runs to the shore, and thence to a long pier where it is dumped on board steamers (Fig. 60).

It has been estimated that the lake still contains 10 to 15 million tons of asphalt. Although vast quantities have been removed in the past, the level of the lake has not sunk more than 8 to 10 ft. since the rate of influx closely approximates the quantity removed. In the past ten years approximately 10 million tons of asphalt have been removed, during which period the level has sunk approximately 1½ meters.

The fresh material consists of an emulsion of asphalt, gas, water, sand and clay. According to Clifford Richardson [25] oil sands occurring at a depth carry an asphaltic petroleum and natural gas under high pressure, which on coming in contact with a paste of colloidal clay and silica are converted into the asphalt which emerges at the surface.

The crude Trinidad Lake asphalt is extremely uniform in composition, as is evident from analyses of samples taken from different points over the surface, calculated on a water- and gas-free basis. The crude material, when freshly sampled at the center of the lake, is composed of:

Water and gas volatilized at 100° C	29.0 per cent
Asphalt soluble in carbon disulfide	39.0 per cent
Asphalt adsorbed by mineral matter	0.3 per cent
Mineral matter on ignition	27.2 per cent
Water of hydration in mineral matter	4.3 per cent
Total	99.8 per cent

Specimens taken from the various portions of the lake's surface, after pulverizing and drying to constant weight in air at room temperature, appear fairly uniform in composition, averaging:

Soluble in carbon disulfide	53.0 to 55.0 per cent
Free mineral matter	35.5 to 37.0 per cent
Water of hydration, etc.	9.7 per cent

The so-called "water of hydration, etc." includes water chemically combined with the clay, asphalt adsorbed by the clay and not removable by carbon disulfide, and the inorganic salts which are volatilized on ignition upon determining the mineral matter.

The mineral constituents consist of extremely finely divided silica and colloidal clay, and have the following composition:

SiO_2	70.68–	66.9 per cent
Al_2O_3	17.04–	20.9 per cent
Fe_2O_3	7.62–	4.4 per cent
CaO	2.46–	1.0 per cent
MgO		1.1 per cent
K_2O and Na_2O	1.22–	1.6 per cent
SO_3	0.97–	3.8 per cent
Cl	0.22–	0.3 per cent
Total	100.00–	100.0 per cent

The mineral matter shows the following granularmetric composition when separated into fractions by means of air:

	Ignited Ash	*Unignited Mineral Residue*
0–10μ	37 per cent	47 per cent
10–20μ	15 per cent	11 per cent
20–30μ	9 per cent	7 per cent
30–40μ	5 per cent	5 per cent
40–50μ	5 per cent	6 per cent
50–60μ	4 per cent	6 per cent
Over 60μ	25 per cent	18 per cent
Total	100 per cent	100 per cent

The mineral constituents of Trinidad asphalt may be separated by treating with bromoform (specific gravity at 77° F. 2.59 to 2.62). The lighter constituents will float to the surface and the heavier constituents present, including titanite, zircon, rutile and glaucophane will settle out. The latter upon examination under the microscope will disclose the characteristic blue color of glaucophane, which although not very plentiful, will aid in the identification of Trinidad asphalt, whenever present in a mixture.[26]

The emulsified water contains mineral constituents in solution to the extent of 82.1 grams (at 110° C.) per kilo, composed largely of sodium chloride. The gas is a mixture of methane, ethane, carbon dioxide, and nitrogen.

The crude asphalt is subjected to a refining process by heating it to 160° C. to drive off the water. A small amount of volatile matter is also removed during this treatment. The refined asphalt has been termed "parianite."[27] A process has been suggested for expelling the water, when the asphalt is to be used for constructing

pavements, which consists in mixing the crude asphalt with a suitable proportion of mineral aggregate heated sufficiently high (e.g., 400° F.) to drive off the water.[28] Another process consists in comminuting the crude asphalt, and drying it at a low temperature to preserve its granular condition.[29] The refined asphalt tests as follows:

(Test 1)	Color in mass	Black	
(Test 4)	Fracture	Conchoidal	
(Test 5)	Lustre	Dull	
(Test 6)	Streak	Black	
(Test 7)	Specific gravity at 77° F.	1.40–1.42	
(Test 9*a*)	Hardness, Moh's scale	1 –2	
(Test 9*b*)	Penetration at 115° F.	10 –15	
	Penetration at 77° F.	1.5 –4.0	
	Penetration at 32° F.	0.25–0.75	
(Test 9*c*)	Consistency at 115° F.	32.7	
	Consistency at 77° F.	74.9	
	Consistency at 32° F.	Above 100	
(Test 9*d*)	Susceptibility index	Greater than 80	
(Test 10*a*)	Ductility (Dow Method):		
	At 115° F.	8.0	
	At 77° F.	1.8	
	At 32° F.	0.1	
(Test 10*b*)	Ductility (Author's Method):		
	At 115° F.	1.5	
	At 77° F.	1.0	
	At 32° F.	0.0	
(Test 11)	Tensile strength (Author's Method):		
	At 115° F.	4.15	
	At 77° F.	21.0	
	At 32° F.	27.0	
(Test 13)	Fraas breaking-point	57° F.	
(Test 15*a*)	Fusing-point (K. and S. method)	188° F.	
(Test 15*b*)	Fusing-point (R. and B. method)	206° F.	
(Test 15*a*)	Fusing-point, *pure* asphalt extracted from mineral matter (K. and S. method)	131½° F.	
(Test 15*b*)	Fusing-point ditto (R. and B. method)	149° F.	
(Test 16)	Volatile at 325° F., in 5 hrs	1.1–1.7	per cent
	Volatile at 409° F., in 5 hrs	4.0–5.25	per cent
(Test 19)	Fixed carbon	10.8–12.0	per cent
(Test 21)	Soluble in carbon disulfide	56–57	per cent
	Asphalt retained by mineral matter	0.3	per cent
	Mineral matter on ignition with tricalcium-phosphate	38.5	per cent
	Water of hydration (clay and silica)	4.2	per cent
(Test 22)	Carbenes	0.0–1.3	per cent
(Test 23)	Soluble in 88° petroleum naphtha (pure asphalt)	62–64	per cent
(Test 26)	Carbon (ash-free basis)	80–82	per cent
(Test 27)	Hydrogen (ash free basis)	10–11	per cent
(Test 28)	Sulfur (ash-free basis)	6–8	per cent
(Test 29)	Nitrogen (ash-free basis)	0.6–0.8	per cent
(Test 33)	Solid paraffins	Trace	

(Test 34*a*)	Saturated hydrocarbons	24.4 per cent
(Test 37*d*)	Saponification value	40.0 per cent
(Test 38*a*)	Free asphaltous acids	6.4 per cent
(Test 38*b*)	Asphaltous acid anhydrides	3.9 per cent
(Test 38*c*)	Asphaltenes	33.0–37.0 per cent
(Test 38*d*)	Asphaltic resins	23.0–26.0 per cent
(Test 38*e*)	Oily constituents	31.0–32.0 per cent

The following more detailed tests have been reported:[30]

Test No.	Description	Refined Trinidad	Extracted Asphalt
7	Specific gravity at 77° F.	1.40	1.070
8*d*	Float test at 212° F.	1492	645
10*a*	Ductility at 77° F.	3	9
13	Fraas breaking-point	66° F.	59° F.
15*a*	Fusing-point (K. and S. method)	171° F.	149° F.
15*b*	Fusing-point (R. and B. method)	203° F.	164° F.
15*h*	Ubbelohde drop-point	230° F.	199½° F.
16	Volatile at 325° F.; 5 hours	0.05%	0.08%
16	Loss (D.I.N. method)	1.10%	1.18%
17*b*	Flash-point (open-cup)	460½° F.	460½° F.
18	Burning-point	529° F.	529° F.
21	Soluble in carbon disulfide	58.62%	99.85%
	Non-mineral matter insoluble	0.20·	0.00
	Mineral matter (ash)	41.18	0.15
	Total	100.00%	100.00%
28	Sulfur	4.65%	5.50%
33	Solid paraffins:		
	Alcohol-ether-fuller's earth	0.25%	0.44%
	Alcohol-ether-sulfuric acid	.....	0.40%
	Alcohol-ether-D.I.N. method	.....	0.28%
37*a*	Acid value	6.0	9.45
38*c*	Asphaltenes	15.35%	26.14%
38*d*	Asphaltic resins	17.25%	29.42%
38*e*	Oily constituents	18.70%	31.90%
	Soft asphalt constituents (diff.)	7.35%	12.54%
	Total	58.65%	100.00%
33	Solid paraffins in oily constituents	0.81%	1.38%

So-called Trinidad "land asphalt" represents material which overflows from the lake at its edges, where it has been exposed to the action of the weather for centuries. It is derived from the same source as the lake asphalt, and has the same general physical and chemical characteristics. It is known under various names; for example: "cheese pitch" is a variety which resembles the lake as-

phalt most closely with respect to its containing gas cavities; "iron pitch" is a variety which has hardened on exposure to the weather to such a degree that it resembles refined lake asphalt; "cokey pitch" is a variety which has been coked or carbonized by brush fires, etc.

The land asphalt varies in its composition from place to place, but differs from the lake asphalt in the following respects:

(1) It contains less gas and water than the lake asphalt.
(2) It contains a slightly higher percentage of mineral matter (from 1 to 2 per cent).
(3) More of the volatile ingredients have been evaporated.

These influence the tests as follows:

The specific gravity is somewhat higher (up to 1.45).
The hardness is greater.
The fusing-point is higher (between 30 and 40° F.).
The volatile matter is less (about 1 per cent).
The percentage of fixed carbon is slightly higher (about 2 per cent).

Brazil

State of Paraná. Asphalt occurrences are reported in many localities in the Sierra da Baliza, in the crevices and pores of the igneous rocks.[31]

State of São Paulo. Asphalt is similarly reported in crevices of a dyke at Fazenda Saltinho, in Rio Tiete, 9½ miles above Porto Martins.[32]

Argentina

Province of Jujuy. An asphalt lake, known as the "Laguna de la Brea," occurs some distance northeast of the City of Jujuy. The asphalt is sulfurous, of a semi-liquid consistency which hardens at the edges. It is mixed with more or less earthy constituents. Seepages of mineral oil are also found locally.

Province of Chubut. Deposits of soft, impure asphalt are reported in the village of Comodoro Rivadavia, associated with seepages of asphaltic petroleum. The asphalt has not been developed, and no analyses are available.

Province of Mendoza. Asphalt deposits, probably resulting from seepages of petroleum, occur in the San Rafael District at Cerro de los Buitres, in the south of Mendoza Province.[33]

COLOMBIA [34]

Department of Bolivar. Numerous seepages of liquid to semi-liquid asphalt, more or less associated with mineral matter, occur along the Caribbean Sea south of Cartagena. These were originally reported by Humboldt in 1788, and have been utilized largely for calking ships.

Department of Antioquia. Numerous occurrences of asphalt have been reported between Nare and Puerto Berrio.[35]

Department of Santander. Similar seepages of soft asphalt, mostly associated with sand and pebbles, occur north of the Sogamoso River which empties into the Magdalena River south of Puerto Wilches. Such asphalts have been reported at Morokoi; at a brook called Puente, a few kilometers north of the foregoing; and still another farther north, at Las Monas.

Department of Boyacá. Deposits of soft asphalt associated with sand and clay are found at Tunja and Sogamoso, north of Bogota, in the Cord Oriental mountain range. These have been used for paving the streets of Bogota. Similar deposits have been reported at Macheta, Tuta, Paipa, Pesca, La Puerta, Topaga, Corrales and San Francisco in the south-central part of this province.[36]

ECUADOR

Province of Guayas. In the Santa Elena peninsula, seepages of asphalt have been reported in pits which have been dug in prospecting oil.[37]

EUROPE

FRANCE [38]

Department of Landes. Near Bastenne, about 24 km. from Orthey, a moderately large-sized deposit of asphaltic sand is found, associated with fossil shells, indicating that this asphalt is of animal (marine) origin. These shells are distributed throughout the asphalt bed, which measures between 10 and 14 ft. thick. On exposure to the air, the shells fall to pieces in a fine powder and the asphalt hardens materially, due to the loss of volatile matter. An analysis by Léon Malo shows the material to contain: asphalt 38.45 per cent, calcium carbonate 4.9 per cent, and sand 56.59 per cent.

This deposit has been worked for many years and was used for constructing the earliest asphalt mastic pavements.[39] Similar deposits were formerly worked at Dax.

Department of Gard. Large deposits of rock asphalt have been mined in the Concession of St. Jean-de-Marvéjols since the first concession was granted by a Royal Ordinance on February 17, 1844. Deposits also occur in the southern part of this Department, including the Concessions of Servas, Cauvas, Les Fumades and Puech. These have long been known (since 1844), but are no longer worked. Lignite and coal occur in the same region. The asphalt is associated with limestone, sandstone and shale, and varies in percentage between 5 and 16 per cent. An average analysis shows it to contain: asphalt 10 to 12 per cent, clay 0.5 to 0.8 per cent, calcium carbonate 84–86 per cent, magnesium carbonate about 2 per cent, and moisture 0.5 per cent. The penetration of the extracted asphalt runs as follows: at 25° C. 98, at 20° C. 58, at 15° C. 32, at 10° C. 20 and at 5° C. 10. At St. Jean-du-Gard it is necessary to sink shafts 1000 ft. deep to reach the stratum of asphalt, which was probably introduced into the limestone as an asphaltic petroleum, the lighter fractions of which evaporated, leaving the asphalt behind.[40]

Department of Haute-Savoie. Deposits of asphalt associated with limestone and sandstone occur at Mussiège, Frangy, Lovagny, Bourbonges, and Chavaroche, in strata between 13 and 16 ft. thick. An analysis of the rock asphalt mined at Chavaroche shows it to contain: asphalt 29.2 per cent, calcium carbonate 51.6 per cent, and sand 19.2 per cent. This is used for paving purposes. The Lovagny deposit contains: asphalt 4.5 per cent, volatile at 100° C. 2.3 per cent, and ash 93.3 per cent. The ash consists of $CaCO_3$ 97.60 per cent, $MgCO_3$ 0.44 per cent, $CaSO_4$ 0.48 per cent, Fe_2O_3 0.62 per cent, and Al_2O_3 0.82 per cent.[41]

Department of Ain. The asphalt deposits extend across the boundary line, separating the Departments of Haute-Savoie and Ain, ranging in a northeasterly direction, and culminating in the Neuchatel (Swiss) region in the north. At Bellegarde, in the northeastern part of the Department, occurs a deposit of asphaltic limestone unevenly impregnated with asphalt, and associated in part with heavy petroleum oils. The well-known Seyssel deposit also

occurs in this Department, at Pyrimont.[42] Minor deposits occur also at Volant, Belley, Challonges, Forens, Obagnoux, Corbonod, Confort, etc., consisting of a fine-grained limestone impregnated with asphalt. This region is illustrated in Fig. 61. The deposits consist of a series of hillside quarries on both banks of the Rhone

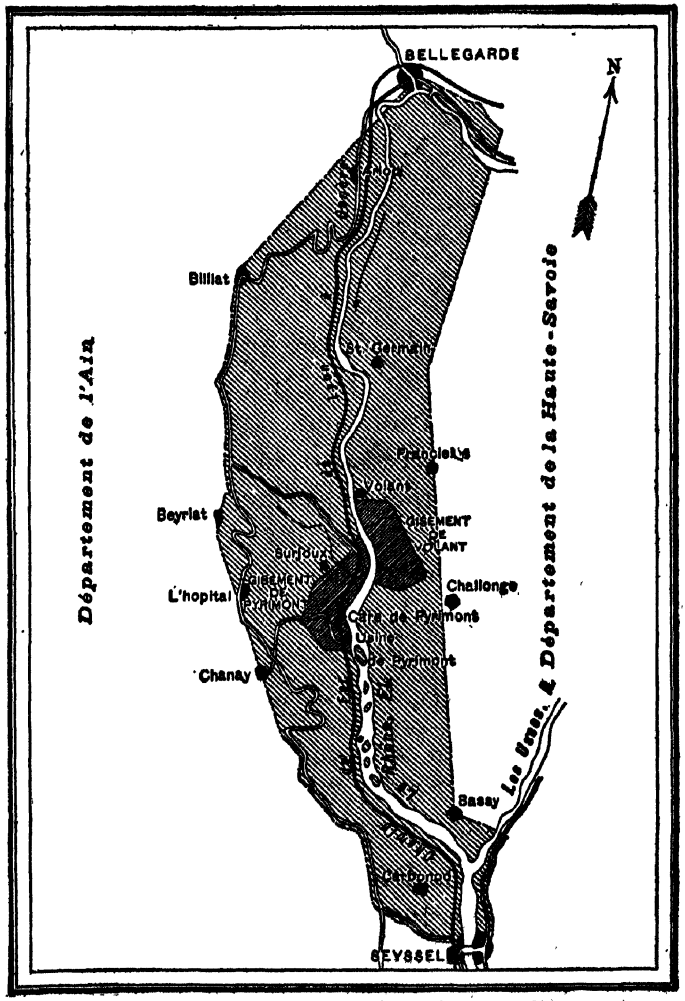


FIG. 61.—Map of Seyssel Asphalt Deposit, France.

River. The asphalt impregnation varies from 2 to 8 per cent as a maximum, the balance consisting almost exclusively of calcium carbonate. Fossil shells, also crystalline calcite, are frequently encountered. The deposits at Pyrimont have been worked for many years by a series of underground tunnels, in eight beds, varying in thick-

ness from 2 to 5 meters, on the eastern bank of the Rhone River. Three grades of asphalt have been produced, containing 3 per cent, 5–6 per cent, and 8 per cent of asphalt respectively. The chief merit of this product lies in the intimacy of its impregnation with asphalt, and the fine granular structure of the associated calcium carbonate. Prior to 1914 from 5000 to 20,000 tons were mined annually. The following analysis represents the average composition of the Seyssel product:

Water	1.9 – 0.0 per cent
Asphalt (fusing-point, 87° F., K. and S. method)	8.00– 8.15 per cent
Magnesium carbonate	0.10 per cent
Calcium carbonate	89.55–91.30 per cent
Iron and aluminium oxides	0.15 per cent
Insoluble in acid	0.10– 0.45 per cent
Loss, etc.	0.10– 0.20 per cent

The extracted asphalt shows the following penetrations (Test 9*b*): at 77° F. too soft; at 60° F. 136; at 50° F. 76; at 40° F. 40.

Fig. 61 shows the location and extent of the Seyssel deposit. Occurrences of asphalt have also been reported at Lelex and Chézery, in the valley of the Valserine, north of Bellegarde.

Department of Basses-Alpes. Two deposits have been described in the vicinity of Forcalquier, one (A) associated with limestone and the other (B) with a siliceous base, testing as follows:[43]

	SiO_2	$CaCO_3$	Fe_2O_3 and Al_2O_3	H_2O and Loss	Asphalt
"A"	Trace to 0.54%	90.63–80.58%	0.01–0.80%	0.13–3.52%	9.33–14.50%
"B"	58.34–59.13%	31.34–28.40%	0.56–0.93%	1.31–0.27%	8.45–11.27%

Department of Puy-de-Dôme (Auvergne). Rock asphalt deposits occur in numerous localities. The only present source of production is at Pont-du-Château (also at Chamalières, near Clermont-Ferrand), where 12,000 to 20,000 tons of rock asphalt composed of 11.0 per cent asphalt associated with SiO_2 (suitable for an acid-resisting mastic) are produced annually. Limited operations have been carried on at various times in the past at Lussat, Malintrat, Gîtes-des-Roys, Puy-de-Croville, du Cortal, Pontgibaud (west of the Chain of Puys), Plain of Limagne, and other minor localities.[44]

Department of Haute Vienne. Minor occurrences have been reported at Limoges, in quartz veins traversing gneiss.

SWITZERLAND

Extensive deposits of asphalt-impregnated limestone occur west of Neuchatel Lake, in the so-called Val de Travers region. These have been exploited for many years and marketed under the names

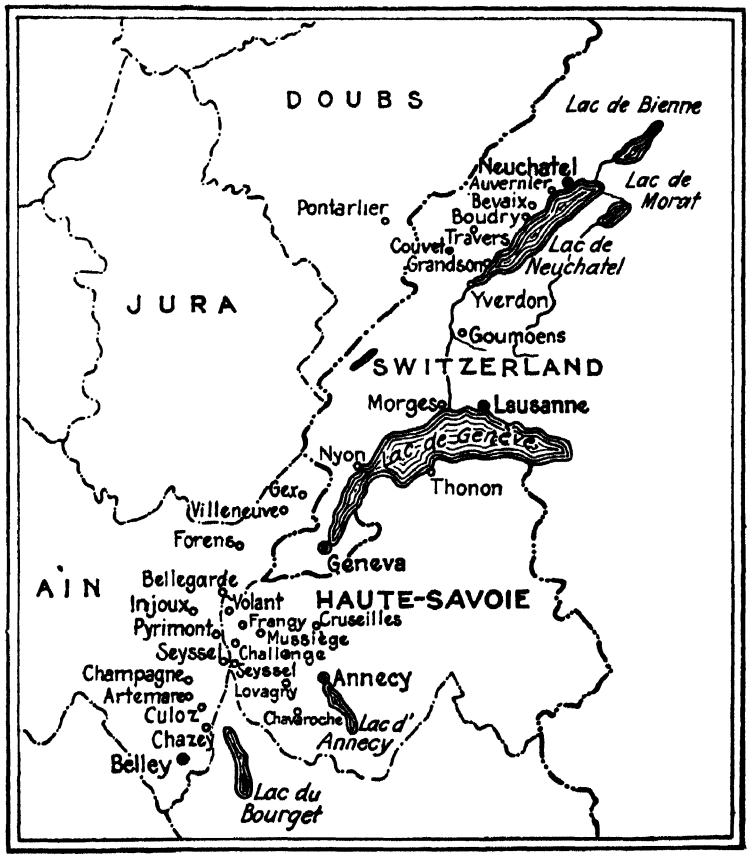


FIG. 62.—Neuchatel and Val de Travers Asphalt Deposits.

"Neuchatel Asphalt" and "Val de Travers Asphalt." [45] The exact location of the region is shown in Fig. 62. The percentage of asphalt varies considerably; thus, the "ordinary" grade contains 8–10 per cent of asphalt, the so-called "rich" grade contains 10–12 per cent of asphalt.

The average product contains: asphalt 10.15 per cent (fusing-point 50° F., K. and S. method), calcium carbonate 88.4 per cent,

calcium sulfate 0.25 per cent, iron and aluminium oxides 0.25 per cent, magnesium carbonate 0.3 per cent, matter insoluble in acid 0.45 per cent, and loss 0.5 per cent. The extracted asphalt shows the following penetrations (Test 9*b*) : at 77° F. too soft; at 60° F. 112; at 50° F. 57. The fusing-point (Test 15*b*) is 43° C., and the ductility at 77° F. (Test 10*a*) over 115 cm.

The theory has been advanced that these asphalts have been produced by the decomposition of marine animal and vegetable matters, which is borne out by the associated fossils.

Outcrops occur at Auvernier, Bevaix, Bois-de-Croix, Presta, Bulles, and St. Aubin, south of Neuchatel, on the western shore of Lake Neuchatel, carrying smaller percentages of asphalt than the preceding.

Alsace-Lorraine

The deposits in this region occur in a well-defined area in the neighborhood of Lobsann a short distance north of Strasbourg, as shown in Fig. 63. The asphalt strata have been traced 6 to 7 miles

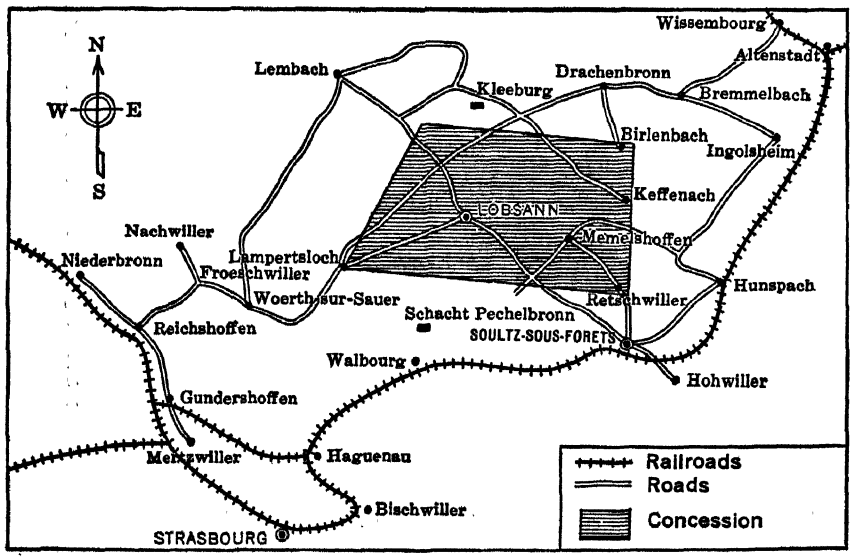


Fig. 63.—Map of Lobsann Asphalt Region, Alsace-Lorraine.

extending through Soultz-sous-Forêts, Pechelbronn and Lampertsloch.[46] They occur as asphalt-impregnated limestone and sandstone associated with lignite. Petroleum is also found locally. The asphalt strata average about 80 ft. in thickness and carry many fossils. The

region is badly faulted. The bituminous limestone has the following average composition:

Asphalt (fusing-point 77° F., K. and S. method)	11.9 –12.32 per cent
Calcium carbonate	69.0 –71.43 per cent
Iron and aluminium oxides	4.3 – 5.9 per cent
Sulfur	5.0 – 5.6 per cent
Magnesium carbonate	0.3 per cent
Silica	3.15– 3.65 per cent
Loss, etc.	1.7 – 3.4 per cent

The asphaltic sandstone at Pechelbronn occurs in veins 3 to 6 ft. thick, containing 15–18 per cent of a soft, viscous asphalt associated with 82–85 per cent of sand. The asphalt when extracted shows a gravity between 0.90 and 0.97 at 77° F. Large quantities of asphalt have been mined in this region for paving purposes.

SPAIN

Burgos Province. At Maesta there has been reported an asphalt deposit containing: 8.80 per cent asphalt, 68.75 per cent SiO_2, 9.15 per cent $CaCO_3$, 8.10 per cent $MgCO_3$, 4.35 per cent Fe_2O_3 and Al_2O_3, and 0.85 per cent water and loss.

GERMANY [47]

Province of Hanover. At Limmer, a small village near Ahlem in the plains of Acker, about 18 miles west of Hanover there occurs a deposit of asphaltic limestone measuring 1600 by 2250 ft. which was discovered by Dr. Eirinis d'Eyrinys about 1730 and first mined in 1843 by D. H. Hennig.[48] The rock carries between 8 and 20 per cent of asphalt and contains numerous fossil shells. As freshly mined it has a brownish to gray-brown color, and the asphalt impregnation is very soft in consistency containing a large proportion of volatile constituents. The average analysis shows:

Asphalt	8.3 per cent
Calcium carbonate	56.5 per cent
Magnesium carbonate	27.0 per cent
Iron and aluminium oxides	8.2 per cent
Total	100.0 per cent

The richer portions of the vein test as follows:

Asphalt (fusing-point 61° F., K. and S. method)	13.4–14.3 per cent
Calcium and magnesium carbonates	67 per cent
Iron and aluminum oxides, etc.	17.5–19.5 per cent
Loss	0.3– 1.18 per cent

At Waltersberge, near Limmer, a very large deposit of asphaltic limestone is found, containing 5 to 7 per cent of asphalt. It is estimated that about 3,000,000 tons occur in this deposit, but the material is so poor in asphalt that it must be enriched before it can be used for paving purposes, although it has not given good results as such, on account of the large percentage of clay present.

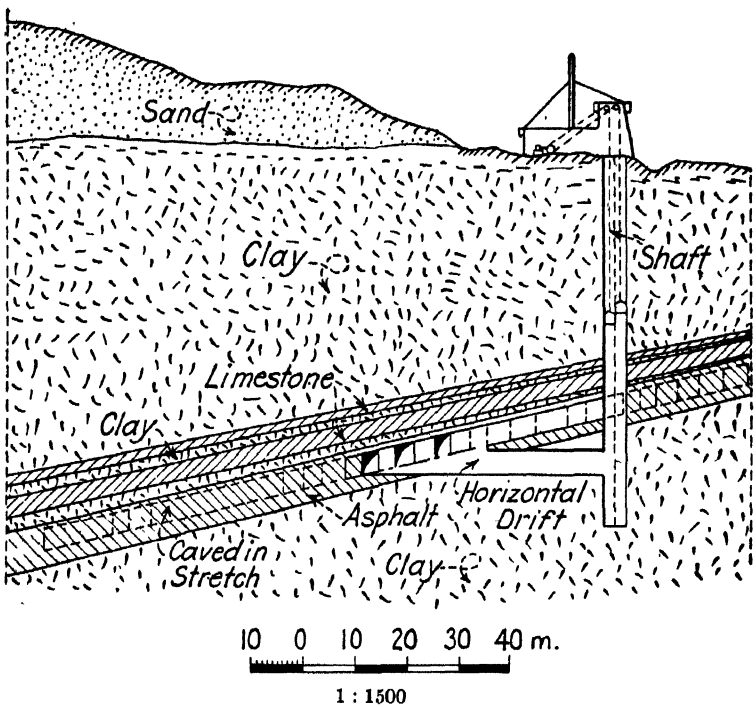


FIG. 64.—Herzog Wilhelm Mine at Holzen, Germany.

In the District of Holzminden, in the Dukedom of Braunschweig, a short distance west of the village of Holzen, on the west bank of the River Ith, there occurs one of the most productive deposits in Germany, which has been identified with the neighboring towns of Eschershausen and Verwohle,[49] discovered in 1601 by woodsmen, who first utilized the substances as fuel. This includes the "Herzog Wilhelm" mine, the "Augusta Victoria," "Verwohle," "Greitbergbruch," "Wintjenberg" and "Stolln Gustav" quarries, the first two of which are illustrated in Figs. 64 and 65. A plan of the entire region is shown in Fig. 66. The deposits consist of as-

phaltic limestone, containing 10 per cent asphalt at the top, which gradually diminishes to 3 per cent at the lower levels, averaging 6 to 8 per cent, and characterized by the presence of fish scales.

The asphalt stratum has been traced for approximately 14,500 ft. and forms a succession of layers 65 ft. thick carrying a variable

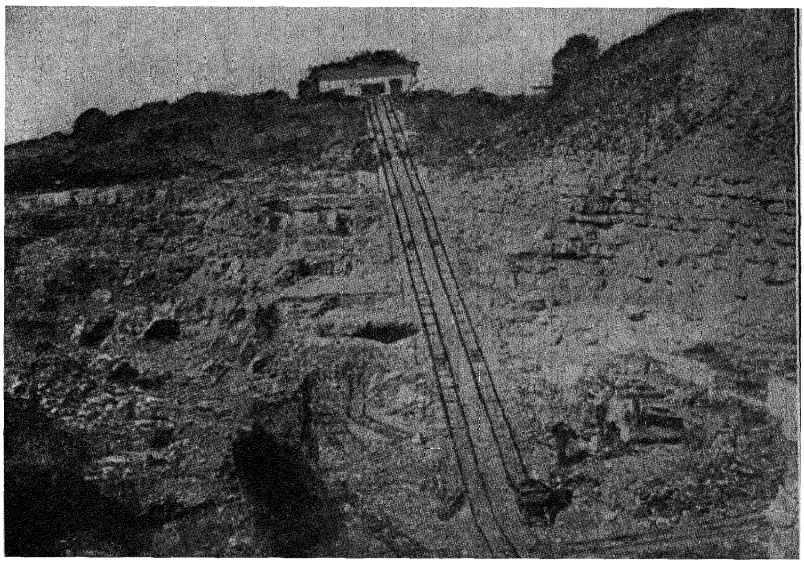


FIG. 65.—Augusta Victoria Quarry at Holzen, Germany.

percentage of asphalt associated with limestone, and separated by clay and shale. The average asphalt analyzes as follows:

Asphalt (fusing-point 65°–70° F., K. and S. method)	5.4 – 8.5 per cent
Calcium carbonate	80.0 –90.9 per cent
Iron and aluminium oxides	4.0 – 5.0 per cent
Silica	2.55– 4.77 per cent
Loss	0.15– 2.11 per cent

The Verwohle rock asphalt shows the following average composition:

(Test 7)	Specific gravity at 77° F.	2.326
	Voids	2.8 per cent
(Test 21)	Soluble in carbon disulfide	7.73 per cent
	Non-mineral matter insoluble	0.60 per cent
	Mineral matter	91.67 per cent

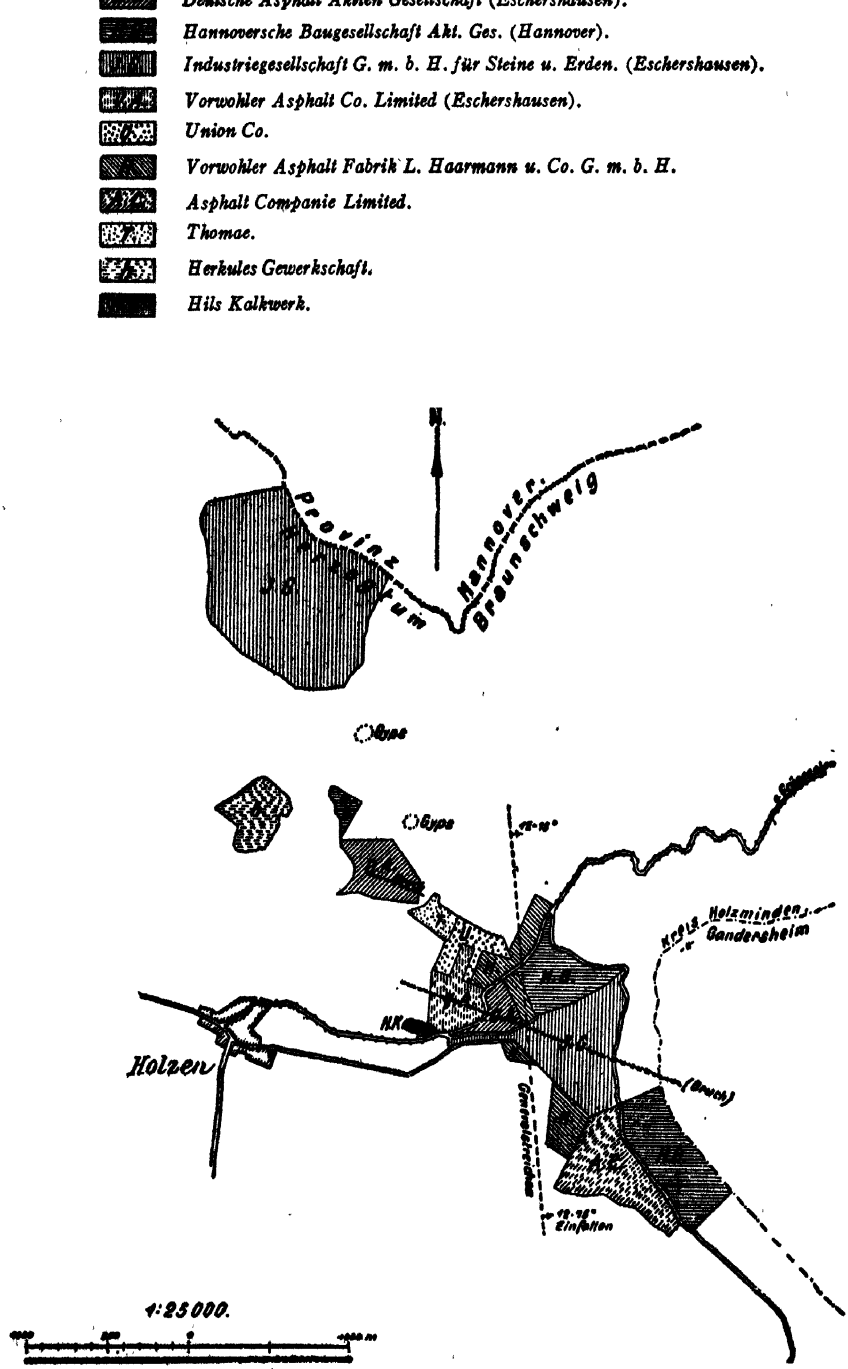


FIG. 66.—Verwohle and Eschershausen Asphalt Deposits, Germany.

The extracted asphalt tests as follows:

(Test 7)	Specific gravity at 77° F	1.019
(Test 9*b*)	Penetration at 77° F	95
(Test 13)	Fraas breaking-point	Below 20° C.
(Test 15*b*)	Fusing-point (R. and B. method)	47° C.
(Test 15*h*)	Ubbelohde drop-point	57° C.
(Test 28)	Sulfur	1.67 per cent

This material must be enriched by the addition of Trinidad, Limmer, or petroleum asphalt, before it becomes suitable for constructing pavements. It is marketed in three forms, viz.:

(1) Mastic cake, which is prepared by first grinding the rock asphalt to a powder, which is heated to 180–200° C. for 4–6 hours, whereupon sufficient asphalt is added to enrich same to 14–16 per cent, and finally cast into the form of cakes, suitable for use in mastic work.

(2) Powder, which is prepared by heating the ground rock asphalt in steam-jacketed kettles to 80° C. with an "enricher" and under strong agitation, until it forms so-called "clinker" containing about 11 per cent asphalt, which in turn is cooled and ground to a powder, suitable for use under compression to form sheet-asphalt pavements.

(3) Asphalt-tiles, which are made by heating the ground rock asphalt and compressing same into plates 28 by 25 cm. and 1.5 to 6.0 cm. thick by means of hydraulic presses operating at 180 to 400 atmospheres. The resulting tiles are used for surfacing factory floors, bridges, viaducts, etc.

A small deposit has been reported at Wintjenberg in this same neighborhood, but has not been worked to any extent.

Province of Westphalia. Minor deposits have been found near the villages of Darfeld, Buldern, Hangenau, and Appelhülsen, associated with clay and shale.

Province of Hessen. At Mettenheim[50] between Worms and Appenheim, occurs a deposit of asphaltic limestone and clay carrying a large quantity of fossil fish remains. The rock contains between 74.4 and 82.6 per cent of asphalt of a comparatively high fusing-point.

Province of Baden. Minor occurrences have been reported near Dossenheim, north of Heidelberg,[51] also near Ottenau, northeast of Baden-Baden.[52]

Province of Silesia. Asphalt has been found in the granite of Striegau and in the northern part of the Black Forest, associated with calcite and hematite.[53]

JUGOSLAVIA

Province of Dalmatia.[54] A deposit of asphaltic limestone occurs at Vrgorac, having a specific gravity at 77° F. of 1.697 containing an average of 26 per cent of asphalt. Analyses show the following ingredients:

	Analysis 1, Per Cent	Analysis 2, Per Cent	Analysis 3, Per Cent
Asphalt	2.94	7.12	38.92
Silica	21.70	.....	.....
Iron and aluminium oxides	7.12	58.10	.....
Iron carbonate	.....	1.10	.....
Calcium carbonate	36.60	.....	61.08
Magnesium carbonate	.....	32.58	.....
Sodium chloride	.....	0.97	.....
Water	4.10	.....	.....
Total	100.00	99.87	100.00

The first analysis represents an asphaltic limestone, containing silica, the second analysis represents an asphaltic shale, and the third an asphaltic limestone (pure). Considerable asphalt has been derived from this deposit for paving purposes.

Asphaltic shales have been reported near the town of Skrip, on the Island of Brazza, situated about 1 mile from the mainland, in the Adriatic Sea, containing between 5 and 40 per cent of soft asphalt, analyzing as follows: asphalt 7.1 per cent, calcium carbonate 58.1 per cent, calcium sulfate 32.6 per cent, silica 2.1 per cent, and undetermined 0.1 per cent. The layers are between 2 and 4 ft. in thickness. There also occurs a deposit of asphaltic limestone containing about 13 per cent of asphalt and 87 per cent of calcium carbonate, having a brownish-black color and containing a substantial proportion of volatile matter.

At Morowitza near Sebenico, on the Adriatic Sea, occurs a deposit of asphaltic limestone carrying 10 to 15 per cent of asphalt, 95 per cent of calcium carbonate, and about 4 per cent of magnesium carbonate.

At Porto Mandorlo, near the town of Trau on the Adriatic Sea, occur beds of crystalline limestone of a brownish color containing 9.2 per cent of asphalt and 90.8 per cent of calcium carbonate. Further deposits have been located in this region at Biskupija and Vinjisće, also at Suhidol, Bua and Dernis-Knin.

Province of Herzegovina.[55] A deposit of asphaltic limestone occurs at the village of Popovo Polje, having a black to grayish-black color, and carrying between 16 to 20 per cent of asphalt. It contains a large percentage of volatile matter, which causes the crude material to ignite very readily and burn with a luminous flame.

A little south of the village of Mišljan, and east of the town of Popovo Polje, occurs another and larger deposit of asphaltic limestone 6 to 20 ft. thick. The asphaltic impregnation is sticky and semi-liquid, varying between 3 and 35 per cent. The richer varieties ignite readily and burn with a luminous, smoky flame.

A deposit of asphaltic limestone about 100 ft. wide and 10 ft. thick occurs at Dračevo, about 2½ miles east of the city of Metkovic. The rock is of a brownish black to dull black color, carrying 5.4 per cent of asphalt. It is not rich enough to be worked profitably.

Province of Styria (Steiermark). Traces have been found at Trenchtling, northeast of Trofaiach, and in the neighborhood of Messnerin.[56]

AUSTRIA

Province of Tyrol.[57] A very peculiar asphaltic shale occurs at Seefeld, 5000 ft. above the sea-level, in beds several feet thick with numerous fossil fish remains, in between layers of dolomite. This deposit constitutes one of the main sources of supply of ichthyol, which is recovered upon subjecting the material to a process of destructive distillation in suitable retorts. The material best suited for this purpose is composed of the following:

Asphalt	26.41 per cent
Calcium and magnesium carbonates	38.22 per cent
Clay	6.67 per cent
Silica	19.03 per cent
Iron oxide	5.95 per cent
Loss and moisture	3.72 per cent
Total	100.00 per cent

HUNGARY [58]

Province of Bihar. Deposits occur at Tataros, Derna and Bodonos, located east and northeast of the town of Nagy-Várad (Grosswardein), between the Sebes Körös and Berettyó Rivers. The Tataros deposit consists of sand containing a soft, sticky asphalt with a characteristic, penetrating odor, which has been exploited under the name "Derna Asphalt." It is associated with a consolidated sandstone in strata between 6 and 25 ft. thick, 5000 ft. long and 4000 ft. wide. Large quantities of asphalt have been mined from this deposit, which constitutes one of the largest sources of supply in Hungary. Analysis shows between 15 and 22 per cent asphalt, fusing at 83° F. (K. and S. method). The water-extraction process has been used to separate a semi-liquid asphalt from the sand, leaving a residue containing 3 per cent of asphalt which could not be separated. The pure, soft asphalt thus separated is distilled to recover the heavy oils and the residue, comprising about 44 per cent of the extracted asphalt, is converted into mastic by mixing with limestone. The product has been used with success for paving the streets of Budapest. During the World War, the purified asphalt was shipped to Germany and used in the manufacture of rubber goods.

A short distance east of Felsö Derna there occurs a bed of sand asphalt, very similar in character and composition to that found at Tataros, carrying 15 to 22 per cent of asphalt. The extracted asphalt contains 0.73 per cent of sulfur, 5.4 per cent of ash, and 1.6 per cent of crystallizable paraffin.[59]

CZECHO-SLOVAKIA

Province of Trecsen. At Strečno, on the River Waag, near Zsolna (Sillein), at the foot of the Lipovec Mountains, there occurs a deposit of dolomitic limestone containing 6.0 per cent asphalt,[60] fusing at 37° C. (K. and S. method). The asphalt contains asphaltic acids 1.37 per cent, asphaltenes 2.11 per cent, asphaltic resins 41.29 per cent, and oily constituents 51.25 per cent.[61]

Provinces of Moravia and Silesia. Occurrences of rock asphalt have been reported at Malenowitz and Zin (northeast of Napagedl), Palkowitz, Chlebowitz (near Friedek), Wiseck (near Lettowitz), etc.

RUMANIA

At Matitza, on both sides of the Matitza River, there occurs a large deposit in veins 30 to 40 m. thick. It is brittle at ordinary temperatures, has a dull, dark color, and a characteristic odor. The material contains 25 to 33 per cent asphalt, having a specific gravity of 1.07 to 1.09 at 77° F., and a fusing-point of 41° C. (K. and S. method). An average analysis shows: water 4.36 per cent, asphalt 30.23 per cent, silica 41.10 per cent, iron and aluminium oxides 18.08 per cent, calcium carbonate 4.43 per cent, and the balance sodium and potassium salts. Large quantities of paraffin wax are produced upon distillation.[62]

ALBANIA

Sélénitza (Sélinitza). A fairly extensive vein of hard asphalt in lenticular form occurs at Sélénitza, 25 km. west of Valona (Avlona), near the junction of the Vojutza (Vojusa) and Sauchista Rivers, close to the railway line. This deposit was first mentioned by Aristotle (384–322 B.C.), and subsequently described in detail by the Roman writer Aelianus Claudius (Aelian), about 100 A.D.[63] It has been used for paving compositions, for manufacturing paints and composition roofings. Between 4000 and 6000 tons are produced annually. It is on the border line between true asphalts and "glance pitch," and tests as follows:[64]

Test No.	Description	Refined Sélénitza	Extracted Asphalt
1	Color in mass	Glossy black	Glossy black
4	Fracture	Conchoidal	Conchoidal
6	Streak	Black	Black
7	Specific gravity at 77° F.	1.23	1.080
9*b*	Penetration at 77° F.	0	0
10*a*	Ductility at 77° F.	0	0
13	Fraas breaking-point	>77° F.	>77° F.
15*a*	Fusing-point (K. and S. method)	230° F.	237° F.
15*b*	Fusing-point (R. and B. method)	264° F.	253½° F.
15*h*	Ubbelohde drop-point	284° F.	295° F.
16	Volatile at 325° F., 5 hrs.	0.0%	0.0%
	Loss (D.I.N. method)	0.6%	0.7%
17*b*	Flash-point (open-cup)	566½° F.	565° F.
18	Burning-point	628° F.	628° F.
21	Soluble in carbon disulfide	84.62%	99.86%
	Non-mineral matter insoluble	0.20%	0.00%
	Mineral matter (ash)	15.18%	0.14%
	Total	100.00%	100.00%

Test No.	Description	Refined Sélénitza	Extracted Asphalt
28	Sulfur	6.25%	7.40%
33	Solid paraffins:		
	Alcohol-ether-fullers' earth	0.59%	0.70%
	Alcohol-ether-sulfuric acid	.....	0.58%
	Alcohol-ether-D.I.N. method	.....	0.40%
37*a*	Acid value	2.35	2.90
38*c*	Asphaltenes	38.20%	45.20%
38*d*	Asphaltic resins	15.90%	18.80%
38*e*	Oily constituents	20.70%	24.50%
	Soft asphaltic constituents	9.72%	11.50%
	Total	84.52%	100.00%
33	Solid paraffins in oily constituents	1.72%	2.03%

ITALY [65]

The general location of the asphalt deposits in Italy are shown in the map illustrated in Fig. 67.

Compartment of Marches.

Province of Pesaro ed Urbino. Impure, solid asphalt is found at Sant' Agata Feltria associated with sulfur, but is not mined actively. At Tallamello and Urbino, deposits of solid and semi-liquid asphalt occur associated with more or less sulfur. These are of no commercial importance, but are of interest merely from a geological standpoint.

Compartment of Abruzzi e Molise.

Province of Chieti. In the neighborhood of San Valentino, extensive deposits of asphaltic limestone have been worked in strata 2½ to 3 miles long and about 100 ft. thick. Quarries have been opened up in the Valley of the Pescara River at the villages of Roccamorice, Abateggio, Manopello, Lettomanopello, Tocco, and Papoli. Three distinct zones are distinguished. The lower carries between 9 and 10 per cent of asphalt, the middle an average of 17 per cent, and the upper 9 to 30 per cent. In certain localities the asphalt has a rubbery consistency, and is deep black in color, and in

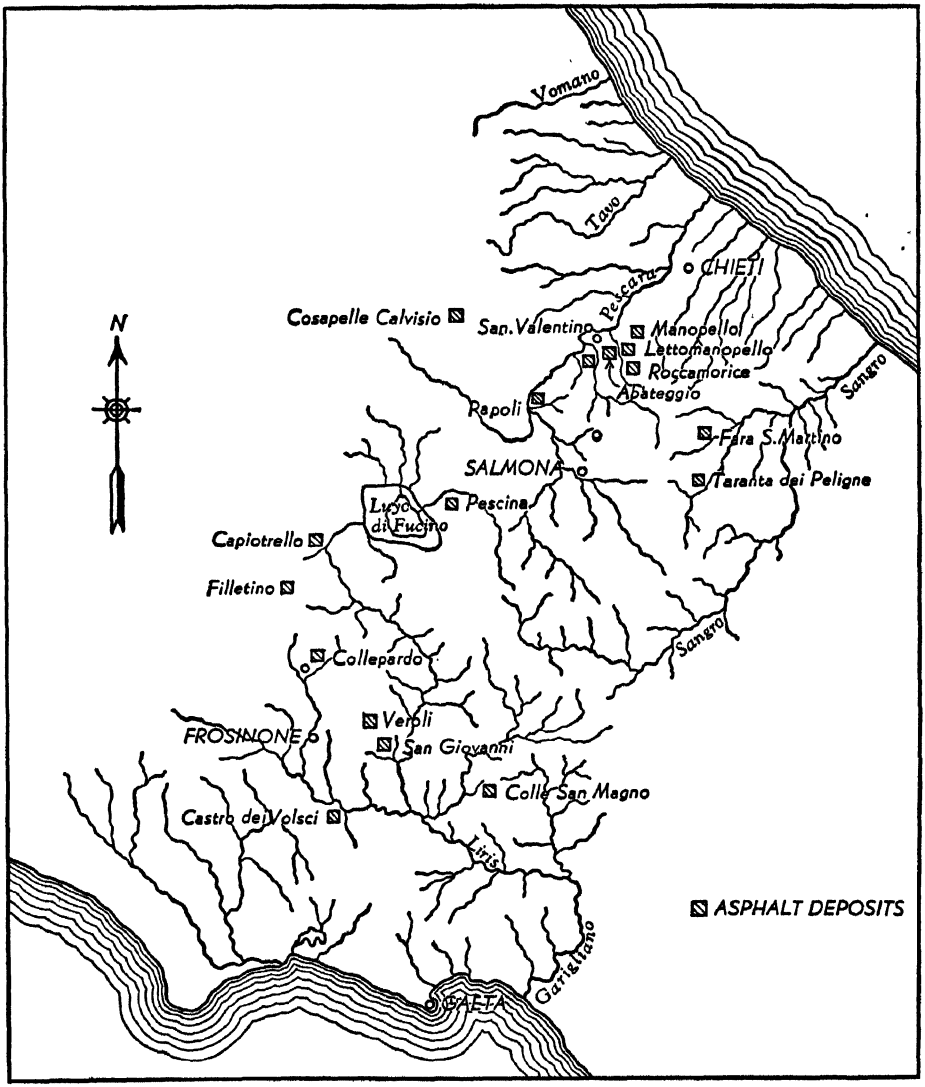


FIG. 67.—Map of Italian Asphalt Deposits.

others it is very soft and semi-liquid. The deposits are rich in fossil shells. Analyses show the following composition:[66]

	San Spirito, Per Cent	Piano dei Monaci, Per Cent	Cusano, Per Cent	San Giorgio, Per Cent	Acqua-fredda, Per Cent	Fonti-celle, Per Cent	Letto-mano-pello, Per Cent	Rocca-morice, Per Cent
Moisture.....	0.64	0.40	0.98	0.46	0.66	0.22	.....	.....
Asphalt.....	10.72	11.70	15.70	12.06	10.62	10.96	7.15	12.46
$CaCO_3$.....	82.25	62.23	49.70	85.30	86.40	86.00	73.76	77.53
$MgCO_3$.....	5.50	24.80	32.00	1.40	1.50	1.20	14.26	4.71
$CaSO_4$.....	.....	.....	.....	.....	.....	.....	1.72	2.63
Clay.....	0.74	0.37	0.32	0.16	0.52	1.18	3.02	2.17
SiO_2.....	0.15	0.06	0.42	0.42	0.30	0.32	0.11	0.50
Loss.....	.....	0.44	0.82	0.20	.....	.....	.....	.....

These deposits are especially suited for pavements in tropical climates and have been used with good results in Cairo, Bombay, Rio de Janeiro, etc. A number of mines have been opened up along this valley amidst precipitous mountains attaining elevations up to 10,000 ft. The rock asphalt is transported by aerial ropeway and narrow gauge surface tracks and the development has been effected in the face of great natural difficulties.

Compartment of Calabria

Province of Basilicata (Potenza). Asphaltic limestone prospects have been reported at Tamutola, Magliano Setere and Leviano.

Compartment of Campania

Province Terra di Lavoro (Caserta). One of the largest asphalt quarries in the entire region, which, however, has not been very active in recent years, occurs at Colle San Magno. Analyses of the product as mined show it to be composed of the following:

Asphalt	7.15 per cent
Calcium carbonate	73.76 per cent
Calcium sulfate	1.72 per cent
Iron and aluminium oxides	3.02 per cent
Magnesium carbonate	14.24 per cent
Silica	0.10 per cent

Prospects of asphaltic limestone occur at Liri, Frosione, Monte San Giovanni, Banco, Castro dei Volsci, Filletino and Collepardo.

Compartment of Sicily

Province of Syracuse.[67] The largest and most important Italian asphalt deposit occurs at Ragusa, about 8 miles from the southern coast of Sicily, on the River Irminio, in a bed 10 to 50 ft. thick and 1600 to 2000 ft. long. It was discovered in 1838 and first utilized for building stone, and later, about 1856, exploited for compressed asphalt work. Fig. 68 shows a general view of the principal mine, Fig. 69 illustrates the tunnelling operations, and Fig. 70 shows the rock asphalt sawn into blocks and slabs as it is blasted from the deposit. The rock contains variable percentages of asphalt, ranging



FIG. 68.—Asphalt Mine at Ragusa, Italy.

from 2 to 30 per cent, associated with a soft consolidated limestone composed largely of fossil shell remains. Several grades of rock asphalt are mined, including a brown variety relatively poor in asphalt, carrying between 3 and 7 per cent, also a black variety carrying about 15 per cent. Commercial grades usually contain 6 to 10 per cent, and occasional pockets are encountered running as high as 12 to 18 per cent. The rock containing 6 to 9 per cent is marketed for use as mastic, and the leaner varieties containing an average of 3 per cent of asphalt are distilled in retorts to recover the oils. In 1924, there were 16 furnaces in operation, yielding 30,000 tons of distillate per annum. The retorts are arranged in groups of four. Each oven is 15.5 meters high and of rectangular

cross section. The waste gases are used to promote combustion of the rock. The maximum temperature is about 730° C. and the distillation zone ranges from 200 to 600° C. The distillate is practically free from paraffin and contains up to 2 per cent phenols. It compares with crude petroleum of Ohio and Texas origin. To

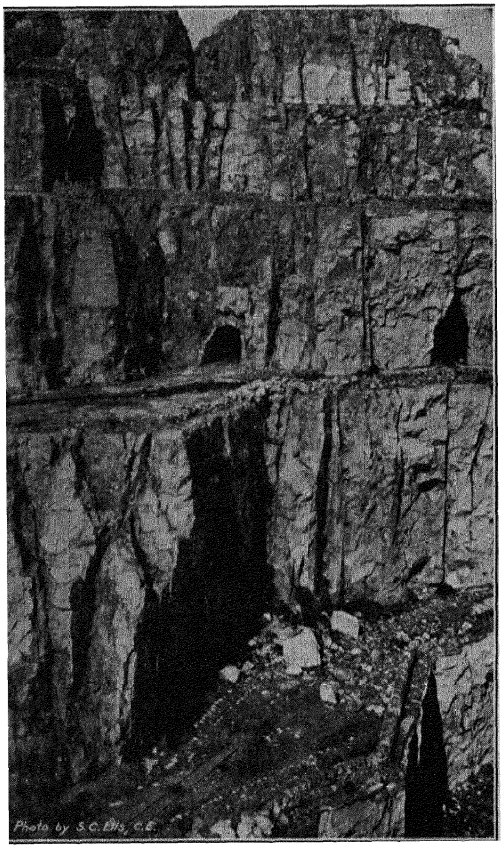


FIG. 69.—Tunnelling Operations at Ragusa Mine.

obtain 1 ton of distillate, 18 to 20 tons of asphaltic rock are required. The distillate is refined with sulfuric acid and fractioned into different grades of lubricating oil.[68] These deposits have been worked for many years, and over 100,000 tons are mined annually for use on the continent of Europe. The material as mined requires no further treatment, other than grinding. It is shipped from the

ports of Siracuse, Mazzarelli and Catania. Analyses show the following compositions:

	Range Per Cent	Average Per Cent
Asphalt	8.80–14.05	9.24
Calcium carbonate	82.15–88.21	87.98
Iron and aluminium oxides	0.91– 1.90	0.93
Magnesium carbonate	0.96	0.72
Silica	0.60– 0.73	0.69
Moisture and loss	0.40– 1.17	0.76

The extracted asphalt shows the following penetrations (Test 9*b*): At 77° F. too soft; at 60° F. 168; at 50° F. 94; at 40° F. 50. The fusing-point (Test 15*b*) ranges between 36 and 44° C.

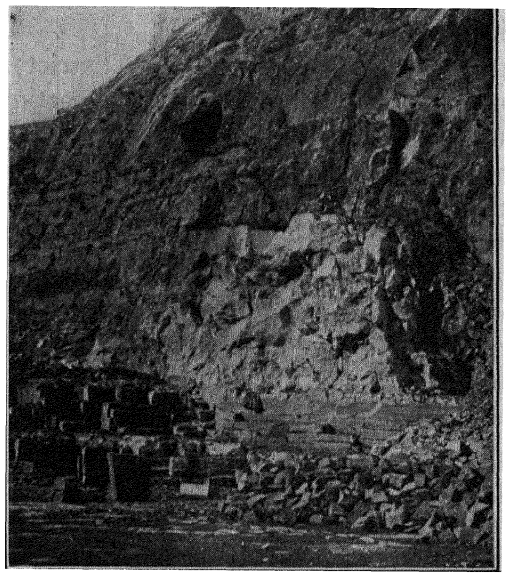


FIG. 70.—Sawing Ragusa Asphalt into Blocks.

The rock asphalt is also removed in large blocks which are capable of being sawed, bored, or carved in the form of paving stones, stair treads, or ornamental work for buildings. The dark color of the asphalt as freshly mined soon disappears upon exposure to the weather, turning to a bluish gray.

Smaller deposits occur at Modica and Scicli, south of Ragusa, in the same region, testing as follows: moisture 0.60 to 0.72 per

cent, asphalt 9.00 to 9.50 per cent, $CaCO_3$ 87.27 to 86.78 per cent, Fe_2O_3 2.05 per cent, SiO_2 0.78 to 0.72 per cent, and loss 0.30 to 0.23 per cent. Asphalt is abundant in the basalt at Cozzo Grillo, near Cape Passero, at the southeastern tip of Sicily, also at Vizzini, north of Ragusa.

GREECE [69]

Department of Triphylia. At the village of Marathonpolis, on the west coast of the Peloponnesus District, there occurs a deposit of asphaltic limestone well suited for the preparation of asphaltic mastic pavements, analyzing as follows: asphalt 14.75 per cent, silica 1.07 per cent, iron and aluminium oxides 0.80 per cent, calcium sulfate 0.21 per cent, magnesium carbonate 0.45 per cent, calcium carbonate 82.27 per cent, moisture and loss 0.45 per cent. This deposit is shown in Fig. 71, location 2.

Department of Achaia (Peloponnesus District). Asphalt has also been reported at location 3, likewise at location 4 (Fig. 71) near Divri, south of Mount Olonos, and north of the town Souli.

Iolian Islands. Liquid asphalt deposits occur on the island of Zante (Zacynthus or Zakynthos) at location 5 (Fig. 71),[70] and asphaltic limestone on the islands of Paxos and Antipaxos, as shown at location 6.

Department of Phocis. A deposit has been reported near Galaxidi, shown at location 7 (Fig. 71).

Department of Phthiotis. Occurrences are found near Drémisa (Dramesi) as indicated at location 8 (Fig. 71).

Departments of Eurytania and Arta. Occurrences of soft asphalt and asphaltic limestone are found in the Pindus Mountains, up to the town of Pinde, as shown in locations 9, 10, 11 and 12 (Fig. 71). Location 11 is at Vordo, in the valley of the Molitsa River, a branch of the Kalamas River.

Northern Departments. Various outcrops of asphalt have been found at Bajousous (location 13), Lavdani (location 14) and Eléousa (location 14), in the Northern Provinces, Fig 71.

PORTUGAL

Province of Estremadura. At Serra de Cabaçoa deposits of asphaltic sandstone have been reported, and at Monte Real, north of Leiria, layers of asphaltic sandstone impregnated with a very

soft and viscous asphalt exist. None of these have been worked to any great extent. An asphalt pit, known as "Azeche," was formerly worked south of Nuestra Senora de la Victoria.[71]

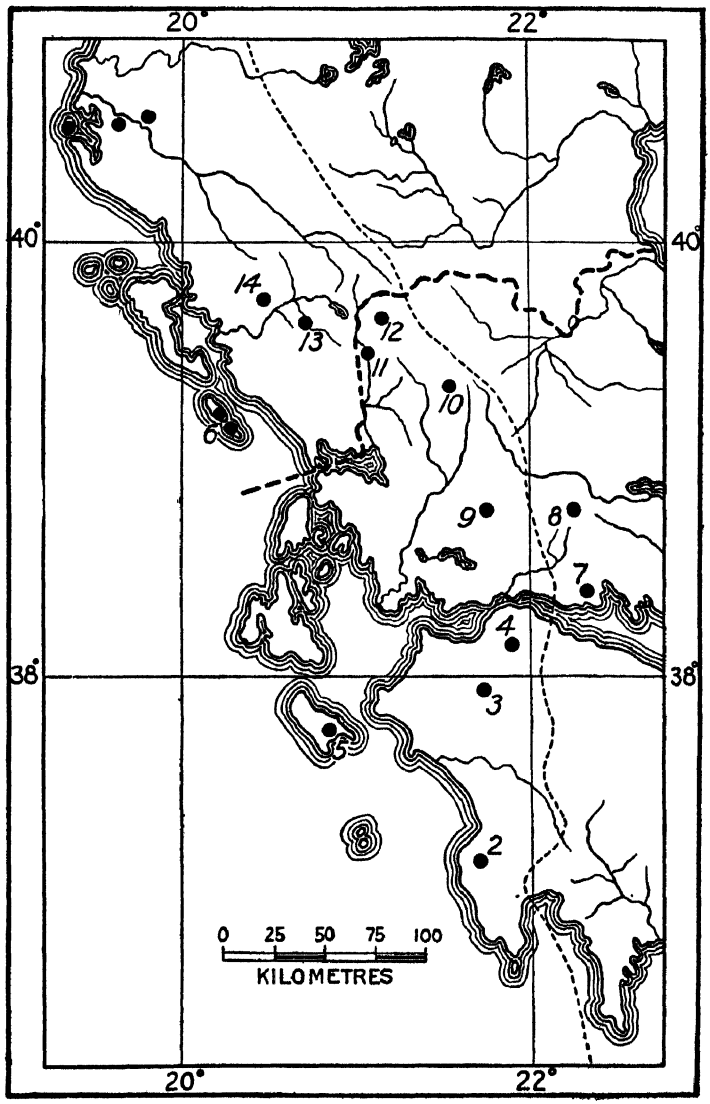


FIG. 71.—Map of Greek Asphalt Deposits.

SPAIN [72]

Province of Santander. In the neighborhood of Puerto del Escudo, deposits of asphaltic sandstone are found in beds about 5 ft. thick. No analyses are available. At Suances similar deposits of

asphaltic sandstone have been reported containing approximately 11 per cent of asphalt, also in the Reviere of Lucara.

Province of Alava. At Alauri, Maestú, and other localities in the Pyrenees, asphaltic sandstone deposits containing 12 to 20 per cent of asphalt have been worked for a number of years. A mine about 10 miles from Vittoria consists of a calcareous sandstone impregnated with 8 to 9 per cent of asphalt. It shows the following average composition:

Asphalt	8.80 per cent
Silica	68.75 per cent
Iron and aluminium oxides	4.35 per cent
Calcium carbonate	9.15 per cent
Magnesium carbonate	8.10 per cent
Water and loss	0.85 per cent

Province of Navarre. Similar deposits have been reported at Bocaicoa, which have been worked to a limited extent.

Province of Gerona. Asphalt and asphaltic shales have been found in the neighborhood of Can Dabano, associated with ozokerite. The asphalt melts at 61.5° C. (K. and S. method).

Province of Tarragona. Asphalt occurs in the District of Campius, in the precipices of Montseng, also at Sot del Bosch, Port Bó, Can Call, Sot de Puig and El Sotas. Likewise in the District Manresa in the Santa Catalina Mountains. Asphaltic shales occur in the District Saedes at Ribas de la Pega, Serrat Negré, Clara and Canal de Dordella.

Province of Soria. At Santander, Sierra de Frentes, and Fuente-toba-Cidones,[73] several deposits of asphaltic sandstone have been operated, from which fairly large quantities have been mined.

Province of Burgos. In the Reviere of Huidobro, asphalt pits have been worked at Narcisa and Felicia.

Province of Almería. Asphalt has been reported at Cobdar, Tíjola and Bayarque.

Province of Valencia. Asphalt has been reported at Mogente.

Russia (in Europe) [74]

Simbirsk Province. The principal occurrences are found along the banks of the Volga River in the vicinity of the estuaries of various small streams, including the Syzranka, extending to Samarskaya-

Louka, between the village Perewoloki and the city Syzran. These contain 6–13 per cent asphalt (rarely 37 per cent) associated with dolomite. In the same region, north of Samarskaya-Louka and 3 km. from the Volga River, there occurs a deposit containing 6–20 per cent asphalt associated with sand, known as "garj," covering an area 1300 by 320 ft., in a layer 32 ft. deep. The extracted asphalt has a fusing-point of 96–147° C.[75]

Kazan Province. Deposits have been reported at Chistopol on the Kama River.

Samara Province. Asphalt is found at the mineral wells at Sergievsk, also at Sarabik-Ulowo and Chugorowo in the vicinity of Bugulma. Deposits are found near Samara, at the hair-pin bend of the Volga, also at Tzarevstchina and on the banks of the Krunza and Oussa Rivers, between Kostyachi and Petcherskoïe.

Terek Province (Northern Caucasia). At Vladikavkaz there occurs a deposit containing 6–12 per cent soft asphalt associated with earthy matter. This is known as "kir" when found in a fairly pure state, and "katran" when associated with much mineral matter. Also 4 km. north of Staniza-Miailovskaja there occurs a deposit in a ravine of the mountain Besimyannya-Gora, containing 70 to 86½ per cent asphalt associated with clay. The extracted asphalt is hard and brittle, melting at about 300° C., and has a specific gravity of about 1.2.[76] Deposits also occur at Goriatchevodsk, likewise on the banks of the Little Chetchna River, between the Terek and Argun Rivers. Asphaltic sands, sandstone and clay occur near the town Sernowodsk. North of the Groznyi oil fields there occurs a deposit containing 6 to 12 per cent asphalt, likewise several small deposits on the Groznyi and Terek rocky prominences in the vicinity of the oil wells.

Kutais Province (Transcaucasia). In the vicinity of Sukhum, on the left bank of Bzyb River, there occur veins of asphalt in dolomite, in the Bsyschro basin. Near Ozurgeti, about 3 km. north of the railroad station Notanebi there occur deposits of asphaltic sandstone containing 20 per cent asphalt. A large deposit occurs 1 km. from the left bank of Supsa River, near where it empties into the Black Sea, to the west of Chrialeti mountain. Deposits are reported near the village Guriamta and asphaltic sands in the vicinity of Tamarasauli, likewise in the valley of Bekwis-Zchali River, near the town

Bekwi, also at the village Dsmuisi about 25 km. northeast of the city Kutais.

Tiflis Province (Transcaucasia). Asphalt deposits occur in the neighborhood of Gori, also the village Dschawa at the overflow of the Big Ljachwa River. Near Signach, about 2 km. northeast of the Mirsaani oil field, in the Kwamianzkaro ravine, there occur four veins of hard asphalt containing 26.3–30.6 per cent ash. Asphaltic sandstones are found on the right bank of the Yora River near Chatma, Kapitschi, and in the rocky prominences of Eilar-Augi near the town Kasaman.

Baku Province (Transcaucasia). Near Baku there are a number of deposits of asphalt in the vicinity of the oil wells, and in the region of the numerous large mud volcanos. In the Adzhikabul (Adza Kabul) district, about 75 km. southeast of Baku, a deposit of rock asphalt has been reported containing 42–67 per cent soluble in carbon disulfide. The extracted asphalt has the characteristics of gilsonite, testing as follows:[77]

(Test 7)	Specific gravity at 77° F	1.026–1.081
(Test 9*b*)	Penetration at 77° F	0 to 9
(Test 15*b*)	Fusing-point (R. & B. method)	196–268° F.
(Test 19)	Fixed carbon	13.6–15.9 per cent

On the Island Suyatoi there is an area of about 14 hektar covered with asphalt, which however is not utilized.

ASIA

Syria (Levant States)[78]

The deposits which follow are indicated on the map shown in Fig. 72.

Vilayet of Aleppo. Various deposits of asphaltic limestone have been reported about 5 miles south of Alexandretta, also near Antakia (Antioch).

Vilayet of Beirut. At Jebel Keferie (Jebel Kfarieh), a hill near the bend of the Nahr el Kabir, near the town Babenna, about 38 km. from the sea, on the road between Latakia and Aleppo, extensive deposits of asphalt are found covering an area of 1400 by 1500 m., estimated to contain about 2,000,000 tons. Most of these consist of a dolomitic marl (containing 28.0 per cent silica, 2.9 per cent $MgCO_3$ and 3.0 per cent clay) with asphalt, of which 21.0 to

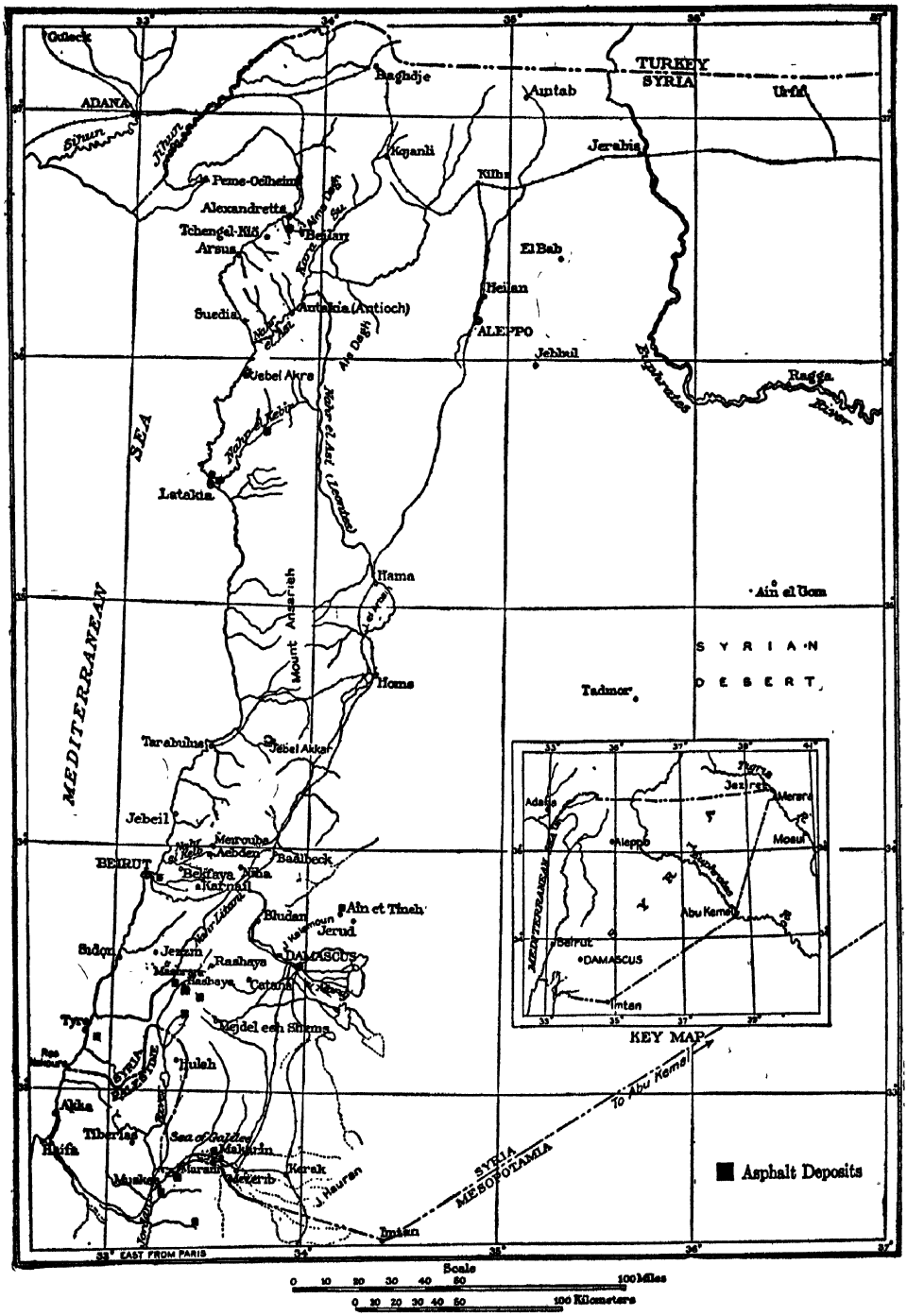


FIG. 72.—Map of Asphalt Locations in Syria and Palestine.

23.6 is soluble in carbon tetrachloride and 10.9 to 10.3 per cent of the non-mineral matter remains insoluble. The bituminous constituents are associated with 68.1 to 66.1 per cent mineral constituents (total = 100 per cent). These deposits have not been worked to any extent, on account of the difficulty in transportation to the coast.[79] They occur in three separate valleys or cañons, i.e., Chandak bu Sfgherie, Chandak Shehade, and Chandak el Barbura; likewise on the neighboring mountains, Jebel Harf and Jebel Nobeh. Similar deposits, but of a smaller size, occur west of the foregoing, and north of a small cañon through which the Nahr el Kebir passes, at Dahana (Derhana, or Derk'ah'a), consisting of limestone and serpentine impregnated with asphalt, of which 10.7 to 12.3 per cent is soluble in CCl_4; also at Milik, consisting of asphaltic marl, of which 6.93 per cent is soluble in CCl_4 and 4.34 per cent non-mineral matter remains insoluble; likewise at Jebel Sullas (Sulas), consisting of limestone and marl impregnated with asphalt, of which 6.99 per cent is soluble in CCl_4. A still smaller deposit lies north of the last three, at Djukr Djak, near Kabara. Still another deposit occurs close to the city of Beirut, and a small one near Tyre (Es Sur). Other deposits are reported at Ain Ibl (Ain Ebel) 20 miles southeast of Tyre; at Aidib (Aideb) 15 miles east of Tyre; and at Hereika.[80]

Vilayet of Sham (Syria).[81] Asphaltic limestone deposits are reported at Khaliwet, between Hasbaya (Hasbeya) and Rashaya (Rasheye), also at localities 5 miles southeast of Hasbaya; another 10 miles south of Hasbaya; another at Sohmor (Sahmur) 10 miles north of Hasbaya on the eastern bank of the Nahr Litani; another at Ain-et-Tineh 7 miles north of Hasbaya on the western bank of the Nahr Litani; and still another 4 miles north of Hasbaya at Ain Tannura (Ain Tadjoura). Asphaltic limestone deposits occur in the Yarmuk (Jarmuk) River valley, along the Damascus-Haifa railroad, at Marani (Mrani) containing 10 to 30 per cent asphalt; also at El-Makarin (Mekarim) containing 18 to 20 per cent asphalt. Rock asphalt also occurs northeast of Latakia, north of Homs, and about 25 miles west of Tyre.

Palestine. Deposits of asphaltic limestone occur at the Sea of Galilee (Lake Gennesaret, or Bahr Tubariya), at Tubariya (Tiberias) and Hammath (Amman, Hamath, or Hammam) on the

western shore, also at Muskes on the River Jordan south of the Sea of Galilee. Asphaltic limestone deposits also occur at the Dead Sea, the largest being at Nebi Musa (Neby Musa) 4 miles west of the northwest shore. This is reported to carry 4 to 6 per cent of asphalt and has a black color. A typical analysis is as follows: asphalt soluble in chloroform 5.07 per cent, $CaCO_3$ 70.20 per cent, $MgCO_3$ trace, $CaSO_4$ 1.20 per cent, SiO_2 4.51 per cent, Al_2O_3 and Fe_2O_3 6.56 per cent, insoluble organic mattter 12.52 per cent, total 100.06 per cent. Other deposits are found on the western shore near Es Sebba (Es Sebbe, or Es Sebeh) and Wadi Sebba (Wadi Sebeh, or Wadi Sebbeth). A short distance from these localities, there occurs a peculiar deposit consisting of flint pebbles cemented together with varying percentages of asphalt, in juxtaposition to a vein of asphaltic limestone. Deposits of asphaltic limestone are also reported on the eastern shore near El Kerak. On the southwestern shore, at the foot of Jebel Usdom (Jebel Esdom) mountain, is found a conglomerate carrying asphalt as the binding medium, and close to this locality, about 300 yards from the mouth of the Wadi Mahawat (Wadi Mayawat) stream there occurs a deposit of asphaltic limestone associated with fossil marine remains, carrying 13 to 25 per cent asphalt, which is used by the natives as fuel.[82]

Mesopotamia (Iraq)

Villayet of Baghdad. At Hit, to the west of Baghdad, on the west bank of the Euphrates River, deposits of asphaltic limestone are still found and collected by the natives in a crude way, exactly as was the case many centuries ago. The occurrences are found a short distance south of Hit, between two streams, the Kubessah and the Mohammedieh. The asphalt is collected and sold in the form of small cakes, which the Turks call "karasakiz" and the Arabs "jir" or "ghir" or "gir"—referring to a mastic containing sand or limestone. The term "seyali" is used to designate the fluid or semi-fluid varieties of asphalt of recent origin, and the term "qasat" to denote the ancient forms of natural asphalt. Upon being melted and mixed with sand or earth, it is used for calking ships. Asphalt deposits 4 to 5 m. thick are found at Tuz-Khurmatli on the west bank of the Tigris River, about 50 miles north of Baghdad. Southwest of this locality there occurs a deposit about 2 m. thick, at the

springs of Kifri, about 2 miles east of the settlement at the foot of Neft-Dagh (Naphtha Mountain).[83] Another deposit occurs on Karum River, north of Dizful, a short distance north of the Persian Gulf. Another deposit has been reported on the southeastern slope of Jebel El-Hamrin mountain along the Tigris River.

Asiatic Russia

Uralsk Province. A deposit of asphaltic sand occurs on the road from Chiwa and Kungrad towards Uil and Uralsky, about 25 km. northeast of the village Tersekan, on the southern shore of a small salt lake.

State of Turkestan. Asphalt deposits occur on the island of Cheleken at so-called Naphtha Mountain (Neftjanaja Gora) and in several other localities, particularly in the vicinity of Ferghana, from which region about 1500 tons of rock asphalt are exported to Europe annually.

Kamchatka Peninsula (Eastern Siberia). On the tundra of the western coast of Kamchatka, north of the River Sopotschnyi, asphalt deposits have been reported.

Sakhalin Island. More or less developed deposits of rock asphalt occur on the northern portion of the island.

Turkey-in-Asia (Asia Minor)

Anatolia. Several minor deposits of rock asphalt have been reported to occur in Anatolia,[84] and at Olty (Olti) in the State of Georgia, but these are of no particular commercial importance.

Arabia

Vilayet of El Hasa.[85] In 1902 an extensive deposit was discovered on the island of Bahrein, in the Persian Gulf, which on analysis was found to consist of:

Asphalt	22.77 per cent
Ash	76.68 per cent
Moisture	0.59 per cent
Total	100.04 per cent

The ash consists almost exclusively of calcium-aluminium silicate. The product is mixed with limestone powder and used for paving

purposes. Another deposit is found near Burgan on the west shore of the Persian Gulf and west of Al-Koweit (Koweyt) near the town of Benaid-el-Qar.

Sinai Peninsula. Asphaltic petroleum seepages occur at Abu Durban and Jebel Tanka.

EGYPT

Springs of asphalt oil have been known as far back as Roman times to exist at Jebel Zeit, termed by them "Mons Petrolius," between the Nile and the Gulf of Suez. Layers of asphaltic sandstone have recently been reported at Helwan, on the right bank of the River Nile, a short distance south of Cairo.[86]

INDIA

Kashmir District. Rock asphalt has been reported near Isakhel on the Basti River.

Hazara District. Seepages of impure liquid asphalt occur in the Sierra Mountains at Gunda.

Baluchistan District. Asphalt seepages similarly occur at Gandava and Khálan (Kháran).

Bombay Island. Deposits of "mineral pitch" and basalt have been reported in Bombay Island, off the western coast of India.[87]

CHINA

Chinese Turkestan (Sin-Kiang). In Sungaria (Dzungaria), asphalt deposits have been reported at Lake Telli-Nor in the Dzung Gobi, southeast of Jair Mountains; also at the Orchu Basin of the Jeun River, on the southern slope of Jair Mountain.

JAPAN

Akita Prefecture.[88] A series of asphalt deposits occur in the villages of Toyokawa (embracing the hamlets of Tsukinoki, Riuge, Magata, Iwase and Urayama) and Kanaashi (embracing the hamlet of Kurokawa). These are situated between 1 to 1½ miles east of the Okybo railway station on the Ōu railroad line, in the neighborhood of Lake Hachiro and the pond Iwase. From the area covered by the deposits, about 180,000 sq. ft., it is estimated there exist a total of 2,360,000 cu. ft. in volume. They are found on the sur-

face of the hills about 30 to 50 meters above sea level, also among the swamps surrounding the hills. It is apparent that this region is underlaid with an asphaltic petroleum which seeps out between the fissures in the shales and flows out of springs in the bottom of the swamps or marshes. For example, in the pond Iwase, the natives stir the bottom with a long bamboo pole, which detaches masses of soft asphalt, which are then ladled from the water's surface. The asphalt occurs in various forms, depending upon the

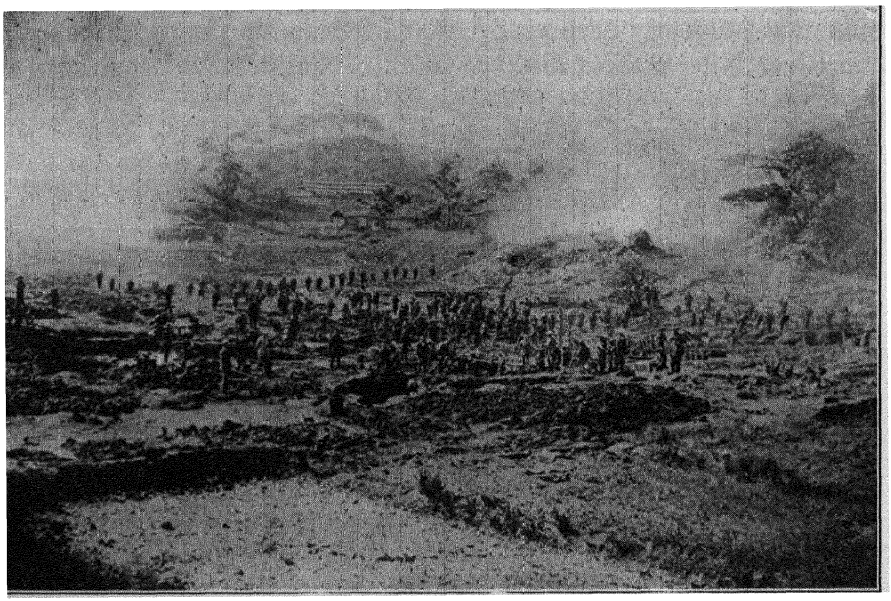


FIG. 73.—Mining Asphalt at Toyokawa, Japan.

state of weathering and the associated ingredients, ranging from a thick viscous maltha to a hard solid form associated with sand or clay, and in swamps with the decayed remains of aquatic plants. As the asphalt exists near the surface of the earth, it is mined by hand with a spade or mattock, as illustrated in Fig. 73. After exposing it to the sun for a while, it is melted in shallow pans to expel the water and permit the coarse earthy matters and organic impurities to settle, whereupon the asphalt is cast into moulds and marketed as such. This operation has been performed in much the same way since ancient times. The good, rich ore is lustrous

and black, whereas the lean, poor ore appears dull brown. Tests made by the author gave the following results:

		Crude Asphalt	*Fluxed Asphalt*
(Test 1)	Color in mass	Black	Black
(Test 2)	Homogeneity	Non-homogeneous	Homogeneous
(Test 4)	Fracture	Conchoidal	Conchoidal
(Test 5)	Lustre	Dull	Bright
(Test 6)	Streak	Brown	Black
(Test 7)	Specific gravity at 77° F	1.280	1.075
(Test 9*b*)	Penetration at 115° F	13	65
	Penetration at 77° F	0.3	15.5
	Penetration at 32° F	0	2.3
(Test 9*c*)	Consistency at 115° F	30.7	10.6
	Consistency at 77° F	83.4	27.6
	Consistency at 32° F	Over 100	66.9
(Test 9*d*)	Susceptibility index	Over 33	31.8
(Test 10*b*)	Ductility at 77° F	0	½
(Test 15*b*)	Fusing-point (R. and B.)	210° F.	154½° F.
(Test 16)	Volatile at 325° F., 5 hrs	2.2 per cent	2.8 per cent
(Test 19)	Fixed carbon	20.7 per cent	20.9 per cent
(Test 21)	Soluble in carbon disulfide	47.72 per cent	86.88 per cent
	Non-mineral matter insoluble	36.85 per cent	9.25 per cent
	Mineral matter	15.43 per cent	3.87 per cent
	Total	100.00 per cent	100.00 per cent
(Test 22)	Carbenes	0.42 per cent	0.12 per cent
(Test 23)	Asphalt soluble in petroleum naphtha	23.2 per cent	60.3 per cent
(Test 33)	Solid paraffins	0.2 per cent	0.5 per cent
(Test 34*a*)	Asphalt as saturated hydrocarbons	10.5 per cent	26.8 per cent

The production ranges from 2000 to 4000 tons annually.

Prefectures of Yamagata, Aomori and District of Hokkaido. Minor deposits have been reported at these localities, but have attained no commercial importance.

AUSTRALIA

New South Wales. Several occurrences have been reported in New South Wales.[89]

Western Australia. Minor deposits have been found in the Ord River basin, 150 miles south of Wyndham, near the junction of the Ord and Negri Rivers; likewise along the Stirling River.[90]

Northern Territory. An occurrence has been reported at the Waggon Lagoon, on Roper River, 350 miles east of Port Darwin.[90]

Tasmania. Asphalt deposits have been reported between Recherche Bay and New River in southern Tasmania.[91]

New Zealand. Asphalt deposits have been located in the Mangawai and Arai Districts, between Bream Tail and Rodney Point.

Dutch East Indies

Buton (Boeton) Island. An extensive asphalt deposit occurs on Buton Island, south of Celebes Island.[92] Illustrated in Figs. 74 and 75. Four different mines have been reported.

(1) Kaboengka, estimated to contain approximately 1,200,000 tons of asphaltic limestone.
(2) Lawele, estimated to contain 100 million tons of asphaltic marl.
(3) Waisioe.
(4) Wariti.

The composition of the asphalt is as follows:

	Kaboengka		Lawele		Waisioe
	(A)	(B)	(A)	(B)	
Crude Asphalt:					
Water	1.5%	2%	2%	10–20%	1.5%
Asphalt	37–39%	26–27%	25–35%	50–60%	35–37%
Organic matter	0.2–1%	0.5–2%	0.5–2%	2–10%	0.2–1%
Mineral matter	58–61%	70–72%	65–75%	10–40%	61–63%
Extracted Asphalt:					
Sp. gr. at 77° F	1.09–1.12	1.06	1.06		1.09
Penetration at 77° F	6–18	65	73		10
Fusing-point (R. and B.)° C.	61–69	51	47		64
Ductility at 77° F	12–110	110	110		32
Insol. in petr. naphtha (88°).	23.7–29.0%	24.5%	25.7%		25.9%

The mineral matter of all the deposits consists of microscopic shells (globigerina)mixed with clay, and has the following composition: $CaCO_3$ 81.62-85.27 per cent; $MgCO_3$ 1.98-3.55 per cent; $CaSO_4$ 0.55-1.70 per cent; CaS 0.17-0.33 per cent; combined water 1.06-2.16 per cent; SiO_2 6.95-9.15 per cent; Fe_2O_3 and Al_2O_3 2.15-3.11 per cent; undetermined 0.32-1.12 per cent.



FIG. 74.—Map of Buton Asphalt Deposits.

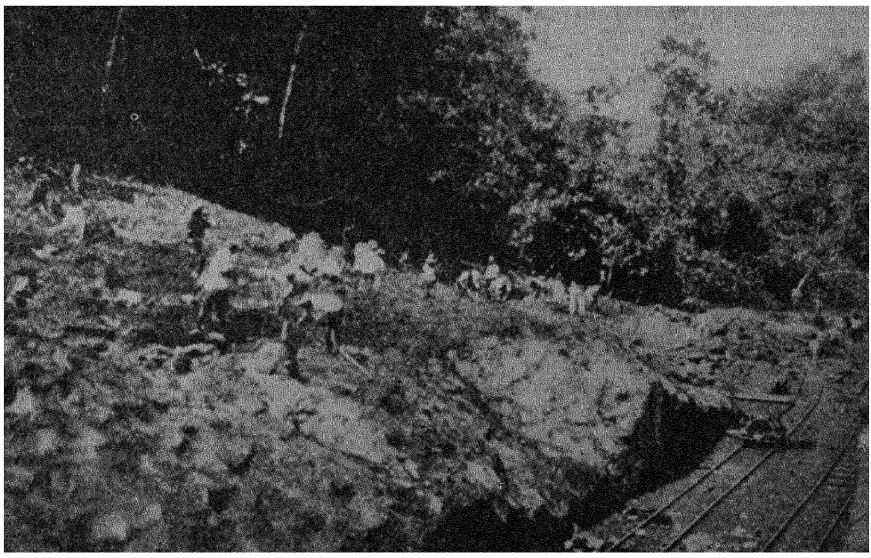


FIG. 75.—Mining Buton Asphalt.

AFRICA

ALGERIA

Province of Oran. At Constantine, asphaltic limestone is found in veins 32 ft. thick, containing as much as 40 per cent of asphalt. In many places the rock is so saturated that the asphalt seeps out and forms pools having a fusing-point of about 140° F. (K. and S. method), a penetration of 11 at 77° F., and containing 0.9 per cent of sulfur. The limestone is largely crystalline. Deposits have also been reported at Sidi Messaoud.[93]

NIGERIA

Bituminous sands are reported in southern Nigeria a short distance from the coast. Attempts have been made to purify the asphalt by the water-extraction process, but so far this has resulted in failure. The extracted material contains about 70 per cent of asphalt, 10 per cent of organic substances other than asphalt and 20 per cent of sand.

RHODESIA

Rock asphalt has recently been reported in northern Rhodesia but is not thoroughly investigated.[94]

MADAGASCAR

Bituminous sandstones containing 3 to 6 per cent asphalt have been reported.[95]

CHAPTER X

ASPHALTITES

Asphaltites[1] are natural asphalt-like substances, characterized by their high fusing-points (over 230° F.). They are grouped into three classes, namely: gilsonite, glance pitch, and grahamite. Since all are presumably derived from the metamorphosis of petroleum, one would naturally expect the classes to merge into one another, and such actually proves to be the case.

The author has adopted the following means of differentiating the three classes, one from another:

	Streak	Specific Gravity at 77° F.	Softening-point (K. and S. Method), Deg. F.	Fixed Carbon, Per Cent
Gilsonite or Uintaite ...	Brown	1.05–1.10	230–350	10–20
Glance Pitch or Manjak*	Black	1.10–1.15	230–350	20–30
Grahamite *.....	Black	1.15–1.20	350–600	30–55

* When substantially free from mineral matter.

In all three classes the non-mineral constituents are almost completely soluble in carbon disulfide. The physical and chemical characteristics will be described in greater detail under the respective headings.

GILSONITE OR UINTAITE

This asphaltite is found in but one region,[2] extending from the eastern portion of the State of Utah across the boundary line into the western portion of Colorado. It occurs in a number of parallel vertical veins, varying in width from thin fissures to several feet.

Gilsonite was first discovered by the early settlers in about 1862, at what was then known as the Culmer vein, several miles south of the present town of Myton in Duchesne County. All of the present veins are vertical and run from northwest to southeast. The gilson-

ite runs fairly uniform in composition, and complies with the following general characteristics:

(Test 1)	Color mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright to fairly bright
(Test 6)	Streak	Brown
(Test 7)	Specific gravity at 77° F.	1.05–1.10
(Test 9*a*)	Hardness on Moh's scale	2
(Test 9*b*)	Hardness, needle penetrometer at 115° F.	3–8
	Hardness, needle penetrometer at 77° F.	0–3
	Hardness, needle penetrometer at 32° F.	0
(Test 9*c*)	Hardness, consistometer at 115° F.	40–60
	Hardness, consistometer at 77° F.	90–120
	Hardness, consistometer at 32° F.	Too hard for test
(Test 9*d*)	Susceptibility index	> 100
(Test 10*a*)	Ductility at 77° F. (Author's Method)	0
(Test 15*a*)	Fusing-point (K. and S. method)	230–350° F.
(Test 15*b*)	Fusing-point (R and B method)	270–400° F.
(Test 16)	Volatile at 325° F., 5 hrs. (dry substance)	Less than 2 per cent
	Volatile at 400° F., 5 hrs.	Less than 4 per cent
	Volatile at 500° F., 5 hrs.	Less than 5 per cent
(Test 19)	Fixed carbon	10–20 per cent
(Test 21)	Soluble in carbon disulfide	Greater than 98 per cent
	Non-mineral matter insoluble	0–1 per cent
	Mineral matter	Trace–1 per cent
(Test 22)	Carbenes	0–½ per cent
(Test 23)	Soluble in 88° petroleum naphtha	10–60 per cent
(Test 26)	Carbon	85–86 per cent
(Test 27)	Hydrogen	8.5–10.0 per cent
(Test 28)	Sulfur	0.3–0.5 per cent
(Test 29)	Nitrogen	2.0–2.8 per cent
(Test 30)	Oxygen	0–2 per cent
(Test 33)	Solid paraffins	0–Trace
(Test 34*b*)	Sulfonation-residue	85–95 per cent
(Test 37*e*)	Saponifiable matter	Trace
(Test 39)	Diazo reaction	No.
(Test 40)	Anthraquinone reaction	No.

Gilsonite is assorted and marketed in two varieties, known as "selects" or "firsts," and "seconds" respectively. The "firsts" are taken from the center of the vein and are characterized by a conchoidal and lustrous fracture. The "seconds" occur near the vein walls and are characterized by a semi-conchoidal and semi-lustrous fracture. They are graded at the mine chiefly by their appearance. The firsts have a higher lustre, a lower fusing-point, a greater solubility in petroleum naphtha, and as produced at the mines run more uniform in physical characteristics than do the seconds. From the middle of the Cowboy vein, there is obtained a variety of gilsonite of unusually high lustre, having a deep black color with a bluish under-

tone, known to the trade as "jet gilsonite," and is used chiefly in the highest grades of baking enamels and japans. It melts about 90° F. higher than the "selects." The "selects" show the least tendency to gelatinize when dissolved in solvents, the "seconds" a moderate tendency, and the "jet" the greatest tendency. The three grades test as follows:

		Selects	Seconds	Jet
(Test 1)	Color in mass.....	Black	Black	Bluish black
(Test 4)	Fracture.....	Conchoidal	Conchoidal	Pencillated
(Test 5)	Lustre.....	Bright	Fairly bright	Very bright
(Test 6)	Streak.....	Brown	Brown	Brown
(Test 7)	Specific grav. at 77° F.	1.044	1.049	1.076
(Test 9*a*)	Hardness (Moh's)....	2	2	2
(Test 15*a*)	Fusing-point (K. and S.)	230–240° F.	240–280° F.	320–350° F.
(Test 15*b*)	Fusing-point (R. and B.)	270–300° F.	275–325° F.	350–400° F.
(Test 23)	Sol. in 88° petroleum naphtha.....	50–60%	20–50%	10–20%

Figure 76 shows the hardness, tensile strength (multiplied by 10) and ductility curves of a mixture of gilsonite and residual oil, fluxed together so as to have a hardness of exactly 25.0 at 77° F.

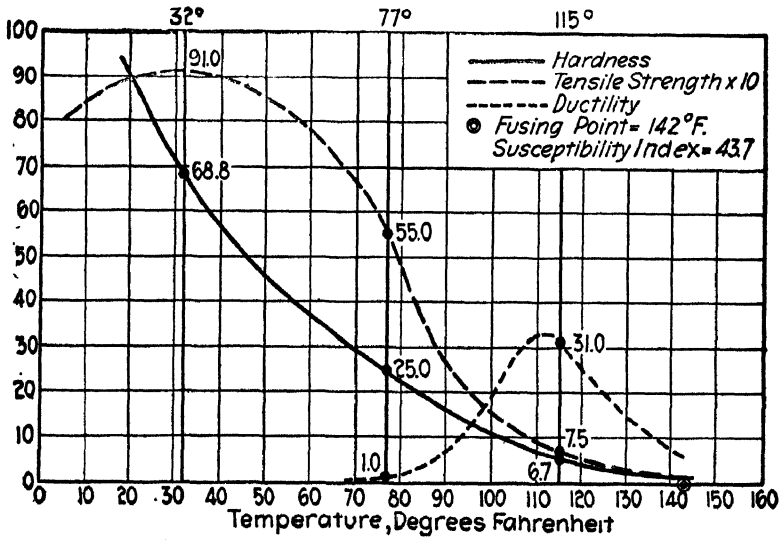


FIG. 76.—Chart of Physical Characteristics of Fluxed Gilsonite.

(Test 9*c*), equivalent to a penetration of 20 at 77° F. (Test 9*b*). The resulting mixture contained gilsonite, 47 per cent and residual oil, 53 per cent. The fusing-point of the gilsonite used was 285° F. (K. and S. method), and that of the resulting mixture 142° F.

Gilsonite is one of the most valuable asphalts for manufacturing paints and varnishes. Gilsonite and glance pitch mix readily in all proportions with fatty-acid pitches, thus differing from grahamite. Products involving the use of gilsonite formed the basis of several patents granted to S. H. Gilson, after whom the material was named.[3]

Gilsonite may be distilled until the vapors reach a temperature of 550° F., when an exothermic reaction takes place and the evolution of gaseous products becomes exceedingly rapid. The source of heat must be reduced at this stage, but when the critical point has been passed, the heating may be continued until the temperature of the solid coke at the bottom of the still reaches 850° F.[4] When the vapor temperature reaches 475 to 650° F. an oily distillate is obtained, which upon refining with sulfuric acid, produces a reddish brown oil suitable for use as an oil substitute in making paints,[5] and the sulfonation product may be used to hydrolyze fats and oils.[6]

NORTH AMERICA

UNITED STATES

Utah.

Uinta County. Practically all gilsonite is mined in the "Uinta Basin," at the junction of the Green and White Rivers south of Fort Duchesne, Utah, from a point 4 to 5 miles within the Colorado boundary line (Rio Blanco County), extending westward about 60 miles into Utah. A large number of veins have been located in this area, extending from a northwesterly to a southeasterly direction, and all of them parallel or nearly so. The veins vary in width from a fraction of an inch to 18 ft., and some of the longest, such as the Cowboy or Bonanza, have been traced 8 miles. The veins are almost vertical with fairly smooth and regular rock walls, and although they are usually continuous, they may in certain cases be interrupted in the direction of the fissure. Very frequently branch veins join the main one, forming very acute angles.

Near the outcrop where it has been exposed to the weather, gilsonite loses its brilliant lustre, changing to a dull black. Along the vein walls it shows a columnar structure, extending at right angles to the wall, which is characteristic of all asphaltites. The

rock walls are often impregnated with gilsonite ½ to 2 ft., so there is no visible line of demarcation between the impregnated and non-impregnated portions. In shale formation the impregnated zone is smaller than when the gilsonite is found in a porous sandstone.

The following are the principal veins occurring in this region:

Duchesne Vein. This occurs about 3 miles east of Fort Duchesne, filling a vertical crack in sandstone and shale. The vein has been traced for 3 miles, and is 3 to 4 ft. wide for about 1 ½ miles, tapering at the ends, until it completely disappears. A comparatively large quantity of gilsonite has been mined from this vein.

Culmer Vein. This is also known as the "Pariette Mine," and occurs in the Castle Peak mining district. It has been traced 7 miles and varies in width from a fraction of an inch to 30 in., averaging about a foot. Several branch veins are connected with the main one at very acute angles. It also shows a number of transverse faults as illustrated in Fig. 77, in which the lateral displacements vary from 1 to 10 ft. The associated rock consists of sandstone and shale.

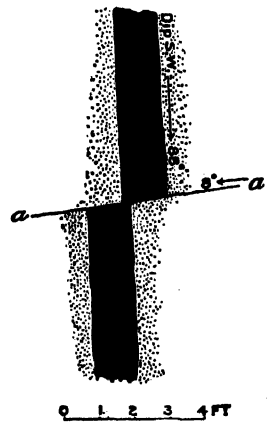
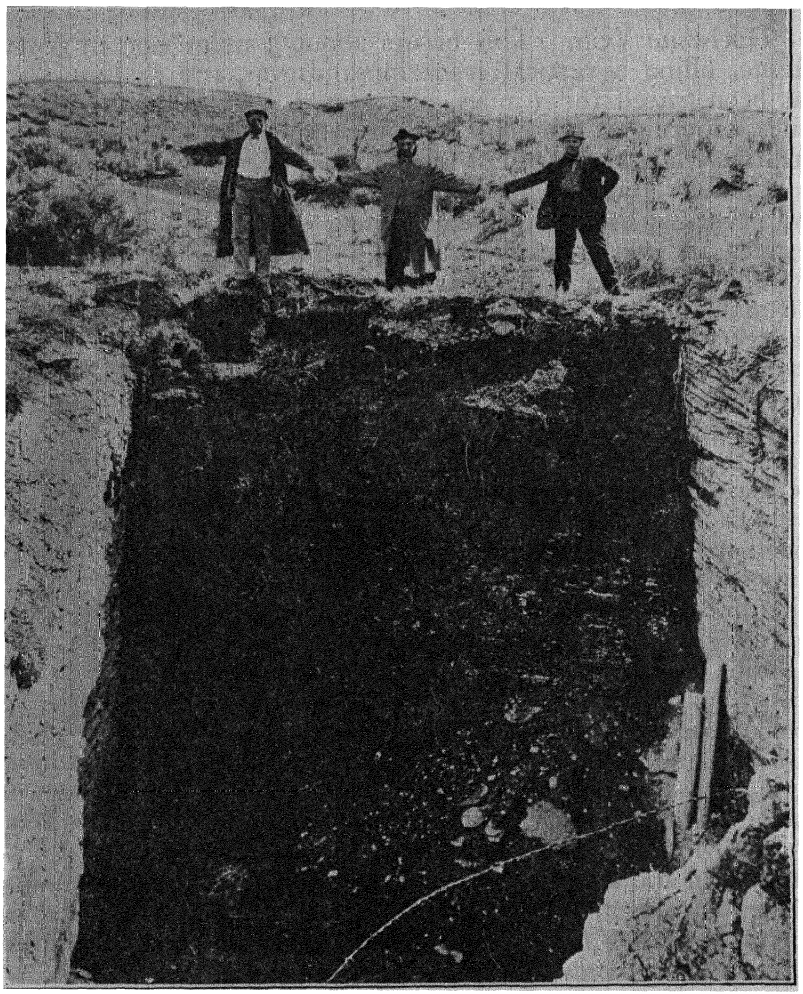


FIG. 77.—Faults in Gilsonite Vein.

Bonanza and Cowboy Veins. These embrace three veins, known as the Cowboy vein, the East Branch and the West Branch respectively of the Bonanza Vein. The last two are joined together near the southern end. These veins occur in sandstone and shale. The shale being harder than the sandstone, seems to have offered greater resistance to the intrusion of gilsonite, and the veins are not therefore as wide when they occur in the latter. The disappearance of the veins to the northwest also occurs in shales, and as the gilsonite passes from sandstone into shale, it splits up into a number of smaller veinlets which gradually thin out into fine hair-like fissures. The Cowboy is the widest vein and attains a maximum width of 18 ft., maintaining for 4 miles a width of 8 to 12 ft., as shown in Fig. 78. Its total length is 7 to 8 miles. The Bonanza veins have similarly been followed for 7 miles, but they are not as wide as the Cowboy.

Dragon group of veins includes the original Dragon vein, the Country Boy and Rector veins, also the Rainbow vein about 8 miles northeast. These occur southwest of Evacuation Creek, a tributary of the White River, near the Colorado boundary line. They extend about 4 miles in length and average between 2 and 3 ft. in width.

In some places the adjacent rock is impregnated with the gilsonite 1 to 3 ft. alongside of the vein proper. In 1911 an explosion shut down the Dragon vein, whereupon the operatives have been transferred to the Rainbow, Country Boy, and Rector veins.



Courtesy of American Asphalt Association.

FIG. 78.—View of Cowboy Gilsonite Mine, Utah.

A number of smaller veins occur in this region, including the Uintah vein, with its branch the Little Emma vein, also the Harrison, Colorado, and other veins.

A branch railroad, known as the Uintah Railroad, about 60 miles long, runs from Dragon, Utah, connecting with the Denver & Rio Grande Railroad at Mack, Col., and then extends to a terminus at Watson, where the Rainbow mine is located. The product from the Country Boy and Rector mines must be trucked 3 to 5 miles,

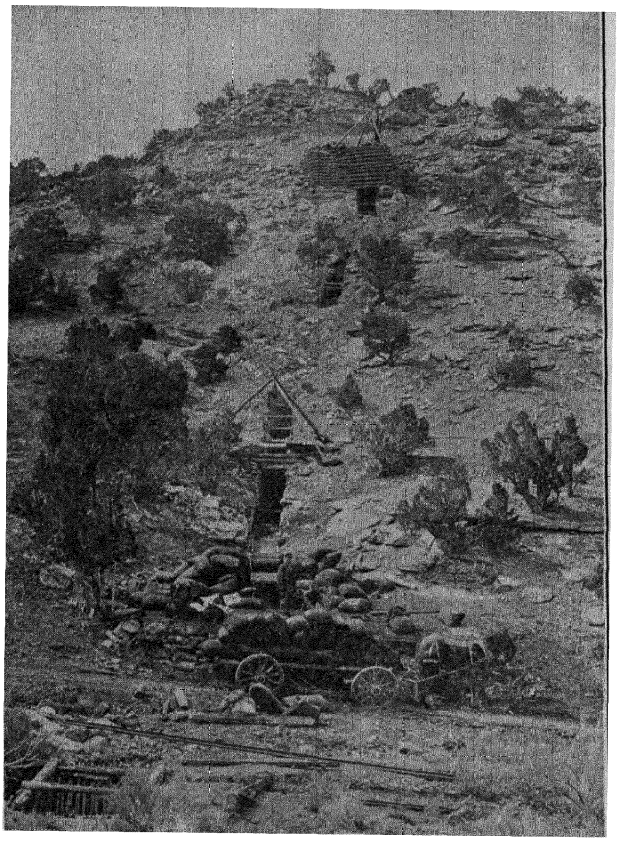


FIG. 79.—Gilsonite Vein, Rainbow Mine, Utah.

and that of the Little Emma mine is trucked to Craig, Colorado. These four mines were the only ones which are operated, producing in aggregate about 130 tons daily. Figures 79 and 80 show two typical views of the Rainbow mine.

The method of mining is very crude, and consists in digging into the veins until a slope of 45 deg. is obtained. The gilsonite is then

dug from the face of the slope, rolled down to the bottom, sacked and hoisted out. The hoisting is done by horses at shallow levels, but when the mine becomes over 200 ft. deep, machinery must be used. Since the ore is brittle, there is always a cloud of dust in the stopes where the men work, which is soluble in the lubricating oil

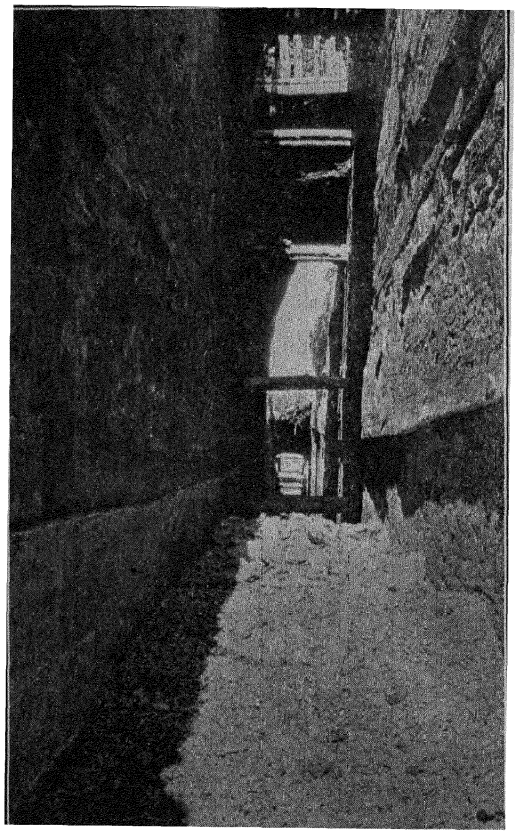


FIG. 80.—Interior of Rainbow Mine, Utah.

used with compressed-air mining tools, thus interfering with the operation of such tools. Experience has therefore shown that the production per man is greater with pick and shovel. One man can mine and sack an average of 2 tons per ten-hour day. Fires are a constant danger, as the dust is highly inflammable, and explosions have been caused by a chain dropping into a pit and striking against the rocky walls, causing sparks.

Very little timber is required, as the veins are nearly vertical, and the surrounding rock is firm and self-supporting. Boulders of rock are frequently encountered in the veins, originally detached from the sidewalls, and prove a serious obstacle. Blasting can only be done where the ventilation is perfect and there is no gilsonite dust, and it frequently becomes necessary to abandon portions of the vein on this account. It is not feasible to work a vein less than 2 ft. wide. It is estimated that approximately 25 million tons of gilsonite are still available in this region.

Oregon.

Wheeler County. Minor deposits of a gilsonite-like material have been reported by E. T. Hodge [7] in the vicinity of Clarno (Type A); also 10 miles east of Post (on the Post-Paulina highway); also on the south bank of Pine Creek near Clarno (Type B), testing as follows:

		Type "A"	*Type "B"*
(Test 1)	Color in mass	Black	Black
(Test 4)	Fracture	Conchoidal	Conchoidal to hackly
(Test 5)	Lustre	Brilliant	Brilliant to dull
(Test 6)	Streak	Brown	Brown
(Test 7)	Specific gravity at 77° F.	1.07–1.08	1.07–1.08
(Test 9*a*)	Hardness	2	2½
(Test 15*b*)	Fusing-point (Note)	525–550° F.	625–650 F.
(Test 16)	Volatile 325° F., 5 hrs.	0–1 per cent	0–1 per cent
(Test 19)	Fixed carbon	5.1 per cent	23 per cent
(Test 21)	Soluble in carbon disulfide	98–99 per cent	75 per cent
	Non-mineral matter insoluble	1–1.5 per cent	24.5 per cent
	Mineral matter	0.5 per cent	0.5 per cent
(Test 22)	Carbenes	1–1.5 per cent	22 per cent
(Test 23)	Soluble in 88° petroleum naphtha	30 per cent	0 per cent

NOTE. The fusing-points are somewhat high for a gilsonite, and approach that of grahamite. The above materials are examples of border-line cases. Both types are undoubtedly similar materials, and Type B has been weathered, so that its original characteristics have been altered. Huntly classes "A" as a gilsonite and "B" as a grahamite.

Crook County. A prospect has been located 38 miles southeast of Prineville, on Sec. 33, T 16 S, R 20 E. (Willamette meridian). It occurs both inside and outside of chalcedony-quartz-calcite geodes, also in the interstices of tuffaceous sandstone, likewise in jasperoid rock alongside a dyke of rhyolite. It appears that the

rhyolite penetrated and fractured an oil-bearing stratum, allowing the oil to ascend along the wall of rhyolite. It tests as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Very bright
(Test 6)	Streak	Reddish brown
(Test 7)	Specific gravity at 77° F	1.162
	Behavior on heating in flame	Softens, froths, and burns
(Test 15*a*)	Fusing-point (K. and S. method)	275° F.
(Test 21)	Soluble in carbon disulfide	83.00 per cent
	Non-mineral matter insoluble	16.39 per cent (Note)
	Mineral matter	0.61 per cent
	Total	100.00 per cent

NOTE. Consists of brown powder, which swells up in carbon disulfide, or when heated.

This asphaltite appears to be a metamorphized gilsonite. Only part is soluble in residual oil (asphaltic), also linseed oil, but on prolonged heating the insoluble matter depolymerizes and gradually goes into solution.

ASIA

RUSSIA

Archangel Province. A hard asphaltite, closely resembling gilsonite, having a specific gravity at 77° F. of 1.0887 and a fusing-point (K. and S. method) of 240° F. has been reported[8] in the Ukhta district on the Izhma River.

GLANCE PITCH

Glance pitch resembles gilsonite in its external appearance, with the exception of the streak, which is a decided brown in the case of gilsonite, and black in the case of glance pitch. It also differs in having a higher specific gravity and producing a larger percentage of fixed carbon. It always has a brilliant conchoidal fracture, and a fusing-point between 230 and 350° F. (K. and S. method). In general, glance pitch complies with the following characteristics:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal to hackly
(Test 5)	Lustre	Bright to fairly bright
(Test 6)	Streak on porcelain	Black
(Test 7)	Specific gravity at 77° F	1.10–1.15

(Test 9*a*)	Hardness, Moh's scale	2
(Test 9*b*)	Hardness, needle penetrometer at 77° F.	0
(Test 9*c*)	Hardness, consistometer at 77° F.	90–120
(Test 9*d*)	Susceptibility index	> 100
(Test 10)	Ductility at 77° F.	0
(Test 15*a*)	Fusing-point (K. and S. method)	230–350° F.
(Test 15*b*)	Fusing-point (R. and B. method)	270–375° F.
(Test 16)	Volatile at 325° F., 5 hrs. (dry substance)	Less than 2 per cent
	Volatile at 400° F., 5 hrs	Less than 4 per cent
(Test 19)	Fixed carbon	20–30 per cent
(Test 21)	Soluble in carbon disulfide	Usually greater than 95 per cent
	Non-mineral matter insoluble	Less than 1 per cent
	Mineral matter	Usually less than 5 per cent
(Test 22)	Carbenes	Less than 1.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	20–50 per cent
(Test 26)	Carbon	80–85 per cent
(Test 27)	Hydrogen	7–12 per cent
(Test 28)	Sulfur	2– 8 per cent
(Test 29)	Nitrogen and oxygen	Trace to 2 per cent
(Test 33)	Solid paraffins	0–Trace
(Test 34*b*)	Sulfonation residue	80–95 per cent
(Test 37*e*)	Saponifiable matter	Trace
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Glance pitch appears to be intermediate between the native asphalts and grahamite. It is probably derived from a different character of petroleum than gilsonite, having nevertheless reached a parallel stage in its metamorphosis, under approximately the same external conditions.

NORTH AMERICA

West Indies

Barbados. Glance pitch was first reported in 1750 by Griffith Hughes, and since 1896 has been mined almost continuously. Deposits occur in a number of localities throughout the island, especially in the Conset district, at Groves, Springfield, St. Margaret, Quinty, and Burnt Hill. This asphaltite has been marketed under the name of "manjak," which was originally applied to the Barbados product, although the name was subsequently associated with a variety of grahamite mined in Trinidad.[9] The deposits were first worked on a commercial scale by Walter Merivale in 1896, who also accurately described the deposit, and the properties of the mineral.

Barbados manjak contains a very small percentage of sulfur (between 0.7 and 0.9 per cent), and about 1-2 per cent of mineral matter. Its specific gravity at 77° F. is in the neighborhood of 1.10,

fusing-point 320-340° F. (K. and S. method), the percentage of fixed carbon as reported by different observers varies between 25 and 30 per cent and its solubility in carbon disulfide 97-98 per cent. Near the surface, the manjak is hard and brittle with a high fusing-point, but at the lower levels of the mines it is found softer, and with a much lower fusing-point, partaking of the nature of an asphalt, rather than an asphaltite, and clearly proving the metamorphosis of one from the other. It also indicates that the manjak originated in the lower strata, having been thrust upward in the form of a dyke (see also Trinidad grahamite).

It is used largely for the manufacture of varnishes and japans on account of its high purity, gloss, and intense black color.[10]

Santo Domingo (Haiti). A deposit of glance pitch similar to the preceding has been reported near Azua on the Bay of Ocoa. This has not been developed commercially, nor are analyses available.

CUBA

There are numerous deposits of glance pitch occurring throughout Cuba, including the following:

Province of Pinar del Rio. Several mines, under the names "Constancia," "San José," etc., occur in the district of Mariel, near the village of Banes, on both sides of the Alfene River, testing as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Brownish black
(Test 7)	Specific gravity at 77° F	1.29–1.34
(Test 9*a*)	Hardness, Moh's scale	2–3
(Test 19)	Fixed carbon	24.3–27.8 per cent
(Test 21)	Soluble in carbon disulfide	70–74 per cent
	Non-mineral matter insoluble	2–3 per cent
	Mineral matter	24–27 per cent
(Test 23)	Soluble in 88° petroleum naphtha	37.5–49.0 per cent

This glance pitch carries considerable adventitious mineral matter, thus differing from the preceding.

Province of Santa Clara. Veins of glance pitch have been reported in the northern portion of Sancti Spiritus district, likewise in Yaguajay district along the eastern bank of River Jatibonico del Norte. The latter are probably a continuation of the veins occurring on the west side of this same river in Province of Camaguey, described below.

Province of Camaguey. In the Moron district, on the west side of River Jatibonico del Norte, several mines of glance pitch resembling gilsonite have been developed, known as "Esperanza," "San Rafael," "Manocal" and "Talaren." The last named is the largest, and the product is shipped from Port Tariffa. The material tests as follows:

		Selects	*Seconds*
(Test 1)	Color in mass	Black	Black
(Test 4)	Fracture	Conchoidal	Conchoidal
(Test 5)	Lustre	Bright	Bright
(Test 6)	Streak	Black	Black
(Test 7)	Specific gravity at 77° F	1.12	1.12
(Test 8*a*)	Hardness, Moh's scale	2	2
(Test 15*a*)	Fusing-point (K. and S. method)	315° F.	345° F.
(Test 15*b*)	Fusing-point (R. and B. method)	284° F.	372° F.
(Test 19)	Fixed carbon	26 per cent	28 per cent
(Test 21)	Soluble in carbon disulfide	99.25 per cent	97.3 per cent
	Non-mineral matter insoluble	0.23 per cent	0.2 per cent
	Mineral matter	0.52 per cent	2.5 per cent
(Test 22)	Carbenes	0.1 per cent	0.2 per cent
(Test 23)	Soluble in 88° petroleum naphtha	18.0 per cent	14.5 per cent
(Test 26)	Carbon	79.7 per cent	....
(Test 27)	Hydrogen	8.2 per cent	....
(Test 28)	Sulfur	7.4 per cent	....
	Undetermined (nitrogen and oxygen)	4.8 per cent	....
	Total	100.0 per cent	....

MEXICO

State of Vera Cruz.

District of Chapapote. As stated previously, deposits of very pure asphalt occur in this locality, varying from very soft consistency to a hard and brittle asphaltite, properly classified as "glance pitch." They show a lustrous and conchoidal fracture, a black streak, fuse in the neighborhood of 250° F. (K. and S. method), contain over 20 per cent of fixed carbon, and are more than 99 per cent soluble in carbon disulfide.

District of Papantla. Glance pitch occurs near the village of Talaxca, about 6 miles north of Papantla, within 30 miles of the port of Gutierrez-Zamora on the Tecolutla River, whence it is transported by lighters to vessels at Tecolutla on the Gulf of Mexico. The glance pitch fills a fissure in hard conglomerate in a vein 2 to 3 feet wide, running north and south for a length of about 35 kilo-

meters. It has been mined to a depth of 80 feet, and tests as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.093–1.108
(Test 15*b*)	Fusing-point (R. and B. method)	298–317° F.
(Test 19)	Fixed carbon	21.5–24.0 per cent
(Test 21)	Soluble in carbon disulfide	99.76 per cent
	Non-mineral matter insoluble	.....
	Mineral matter	0.24 per cent
	Total	100.00 per cent
(Test 22)	Soluble in carbon tetrachloride	99.72 per cent
(Test 23)	Soluble in 88° petroleum naphtha	27–35 per cent
(Test 28)	Sulfur	7.13 per cent

UNITED STATES

Utah.

Emery County. Deposits of glance pitch occur at Temple mountain, 45 miles southeast of the town of Greenriver on the eastern flank of San Rafael swell, also at Flat Top mountain, 2½ miles to the southeast. Occur as glistening black nodules ranging in size up to 2½ in. in diameter in a bed of sandstone conglomerate about 60 ft. thick. The asphaltite is characterized by the fact that it carries uranium and vanadium, which are assumed to have been incorporated in it during its migration from the underlying strata, thereby hardening the asphaltite and "fixing" it in the present associated rocks. The material contains an average of 1.75 per cent U_3O_8 and 4 per cent V_2O_5. Specimens taken from the Cowboy claim (Shinarump conglomerate), designated "A," and from the lower part of sandstone on the west slope of Temple mountain, designated "B," analyze as follows:

	Specimen "A," Per cent	*Specimen "B," Per cent*
Loss on ignition	49.57	67.11
Ash soluble in HCl:		
Uranium oxide (U_3O_8)	1.13	2.88
Vanadium oxide (V_2O_5)	0.23	1.17
Sulfur	4.98	1.37
Arsenic	Trace	Trace
Selenium	None	None
Ash insoluble in HCl	46.32	26.54
Total	102.23	99.07

In many places the asphaltite has completely weathered away, leaving deposits of the metals and metalloids in the form of uranium and vanadium hydroxides, uranium vanadate, calcium vanadates (i.e., red and green varieties), calcium-uranium vanadate, potassium-uranium vanadate, copper-uranium arsenate, etc.[11]

CENTRAL AMERICA[12]

NICARAGUA

District of Chontales. A vein of glance pitch has been reported about 10 miles northeast of Santo Tomas, northeast of the mountain range, about 1 in. thick, in a fault plane of small displacement, cutting water-laid volcanic tuffa.

SALVADOR

Department of San Miguel. At the waterfall in Quebrada Granda, one mile southwest of the "Otuscal Ranch," glance pitch is present as a fracture filling, associated with calcite and chalcedony.

SOUTH AMERICA

COLOMBIA

Department of Tolima. A large deposit of glance pitch occurs at Chaparral on the Saldaña River, which empties into the Magdalena River. The deposit is about 100 miles southwest of Bogota. It is transported by boats down the Magdalena River to the coast, whence it is exported. About 2000 tons are shipped annually having a high fusing-point and over 99 per cent soluble in carbon disulfide.[13] It is used largely for the manufacture of varnish and tests as follows:

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.12
(Test 8*a*)	Hardness, Moh's scale	2
(Test 8*b*)	Hardness, penetrometer	0
(Test 15)	Fusing-point (K. and S. method)	275° F.
(Test 19)	Fixed carbon	26.45 per cent
(Test 21)	Solubility in carbon disulfide	96.0 per cent
	Non-mineral matter insoluble	0.7 per cent
	Mineral matter	3.3 per cent

Department of Bolivar. A similar deposit occurs at Simiti, on the western bank of the Magdalena River, north of the foregoing deposit.

EUROPE

GERMANY

Bentheim. Deposits of asphaltite, probably of the nature of glance pitch, are found in the vicinity of Bentheim. These were first regarded as a coal, and have been worked since 1732. The material is hard, shows a glossy, conchoidal fracture, a black streak, specific gravity of 1.07 to 1.09, and 0.53 to 11.24 per cent ash.[14]

ASIA

SYRIA (LEVANT STATES)

Vilayet of Sham (Syria). An extensive series of glance pitch deposits occur in the vicinity of Hasbaya (see map, Fig. 72) in the upper Jordan valley, on the western slope of Mount Hermon (Jebel esh Sheikh). The principal mine, known as "Sûk el Chan" or "Bir el Hummar" is located on the eastern slope of Jebel ed Dahr, a hill separating the Jordan River (Nahr Hasbani) and the Nahr Litani, where a deposit of glance pitch of brownish black color is found in a vein up to 4 m. thick. It has a brilliant lustre and a black streak, specific gravity at 60° F. of 1.104, contains from a trace to 5 per cent mineral matter, fuses at 275° F., and yields 27 per cent fixed carbon. An average specimen on analysis shows: carbon 77.18 per cent, hydrogen 9.07 per cent, sulfur 0.40 per cent, nitrogen 2.10 per cent, and mineral matter 0.50 per cent. This deposit is said to have been worked since about 1600 B.C. Some of the ancient pits are still to be seen, as deep as 60 m. From 1890 to 1900, about 66,000 tons were mined, and a quantity exported to the United States for the manufacture of varnishes, for which it is well suited. In this same region there is found an asphaltic limestone impregnated with an average of 10 per cent asphalt of comparatively high fusing-point (presumably glance pitch) associated with petrified fish remains.[15]

Palestine. The Dead Sea deposits are merely of historical interest, as they constituted one of the principal sources of supply of asphalt for the ancients. There appear to be large veins of asphalt at the bottom of the Dead Sea, the water of which is saturated with salt (25 per cent solution) having a gravity of about 1.21. The asphalt has a specific gravity at 77° F. of 1.104, and as masses become detached at the bottom by earthquake shocks or otherwise, they float to the surface, where they are gathered up by natives. A section through the Dead Sea showing the veins of asphaltite is illustrated in Fig. 81. Deposits also occur at Es Sebba (Es Sebbe)

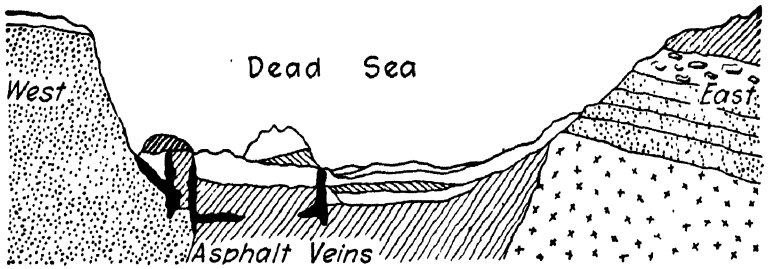


FIG. 81.—Vertical Section through Dead Sea Showing Glance Pitch Veins.

and Masada on the west shore, and at Seil-el-Modschib on the east shore of the Dead Sea. This glance pitch shows a lustrous, conchoidal fracture, and a black streak. Its fusing-point is 275° F., over 99 per cent is soluble in carbon disulfide, and it yields 20 per cent of fixed carbon. The supply is limited and the material is used only to a small extent locally.[16]

MESOPOTAMIA (IRAQ)

A deposit occurs at Abu Gir on the Euphrates River, west of Baghdad, containing 86.5 per cent soluble in carbon disulfide, 10.0 per cent mineral matter and 3.5 per cent water; having a fusing-point (R. and B.) of 172° C. and containing 7.3 per cent sulfur. The mineral constitutents contain Ca, Mg and Fe, with a trace of vanadium.

GRAHAMITE

This asphaltite varies considerably in composition and physical properties, some deposits occurring fairly pure and others are asso-

ciated with considerable mineral matter, running as high as 50 per cent.[17] In general, however, it complies with the following:

Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal to hackly
(Test 5)	Lustre	Very bright to dull
(Test 6)	Streak on porcelain	Black
(Test 7)	Specific gravity at 77° F.:	
	Pure varieties (containing less than 10 per cent mineral matter)	1.15–1.20
	Impure varieties (containing more than 10 cent mineral matter)	1.175–1.50
(Test 9*a*)	Hardness, Moh's scale	2–3
(Test 9*b*)	Hardness, needle penetrometer at 77° F.	0
(Test 9*c*)	Hardness, consistometer at 77° F.	Over 150
(Test 9*d*)	Susceptibility index	> 100
	Behavior on heating in flame:	
	Variety showing a conchoidal fracture and a black lustre	Decrepitates violently
	Variety showing a hackly fracture and a fairly bright to dull lustre	Softens, splits and burns
(Test 15*a*)	Fusing-point (K. and S. method)	350–600° F.
(Test 15*b*)	Fusing-point (R. and B. method)	370–625° F.
(Test 16)	Volatile at 500° F., 5 hrs	Less than 1 per cent
(Test 19)	Fixed carbon	30–55 per cent
(Test 21)	Soluble in carbon disulfide	45–100 per cent
	Non-mineral matter insoluble in carbon disulfide	Less than 5 per cent
	Mineral matter	Variable (up to 50) per cent
(Test 22)	Carbenes	0–80 per cent
(Test 23)	Soluble in 88° petroleum naphtha	Trace–50 per cent
(Test 30)	Oxygen in non-mineral matter	0–2 per cent
(Test 33)	Solid paraffins	0–Trace per cent
(Test 34*b*)	Sulfonation residue	80–95 per cent
(Test 37*e*)	Saponifiable matter	Trace
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

In general, grahamite is characterized by the following features:

(1) High specific gravity;
(2) Black streak;
(3) High fusing-point;
(4) High percentage of fixed carbon,
(5) Solubility of non-mineral matter in carbon disulfide.

A process has been proposed for reducing the fusing-point of grahamite, by heating the material either alone, or with a proportion of semi-asphaltic residual oil (12 to 14° Baumé) in a closed retort at 400° F. for twenty-four hours, under a pressure of 50 lb. per sq. in. This converts the asphaltite into a product similar in

appearance to gilsonite, having a black streak. The percentage of carbenes is reduced and the proportion soluble in 88° petroleum naphtha is increased.[18] Processes have been described for fluxing grahamite with coal tar [19]; also for fluxing grahamite and adding a mineral filler.[20]

Deposits of grahamite occur in the following localities:

NORTH AMERICA

UNITED STATES

West Virginia.

Ritchie County. The original deposit of grahamite was discovered in West Virginia.[21] It was first described by Prof. J. P.

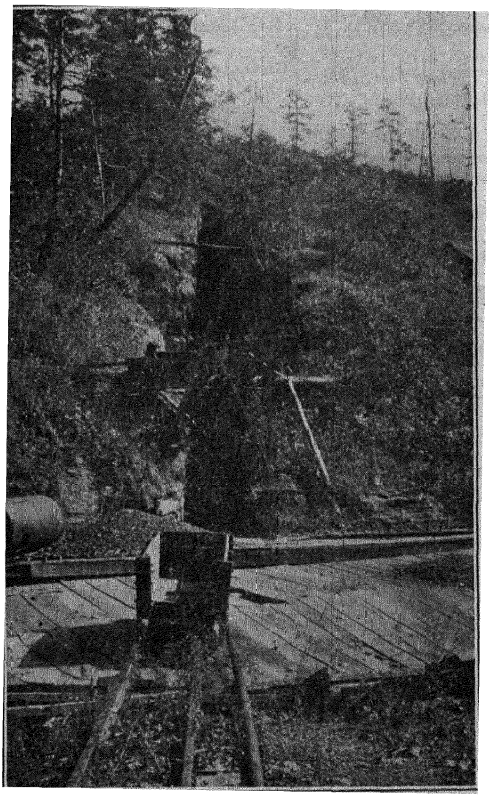


FIG. 82.—View of Grahamite Vein, Ritchie County, West Virginia.

Leslie in a paper read before the American Philosophical Society, March 20, 1863. It is found in but a single locality in Ritchie County, about 25 miles southeast of Parkersburg. The grahamite fills an almost vertical fissure in sandstone, a mile long, varying in width from 2 in. at the ends to 4 and 5 ft. in the center. Its depth is assumed to be 1500 to 1600 ft.

The mine has long been abandoned, as the available supply of grahamite is exhausted. Figure 82 shows a view of the opening in

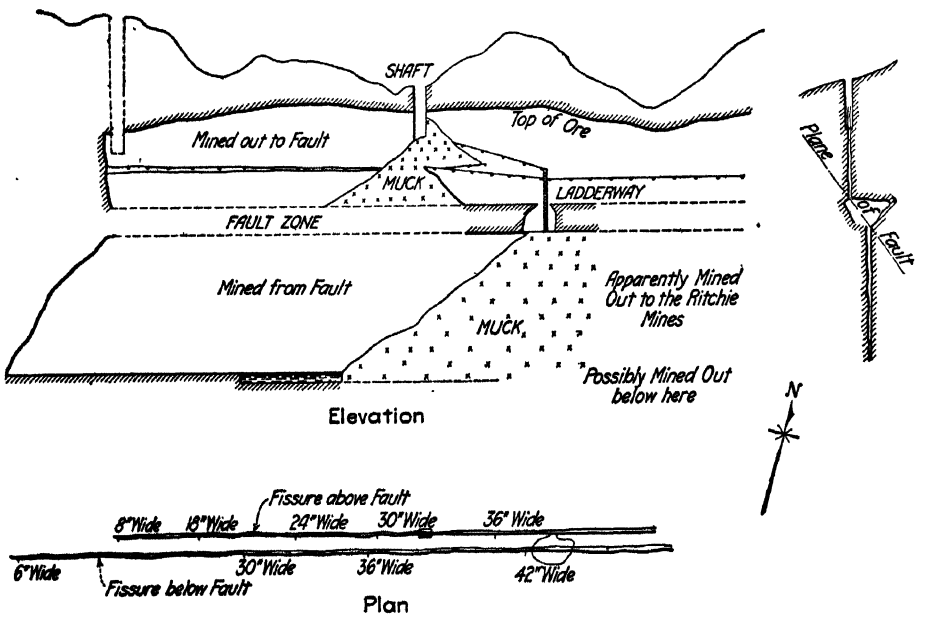


FIG. 83.—Sections through Grahamite Mine, Ritchie County, W. Va.

the hillside from which the grahamite has been removed. Figure 83 shows the nature and extent of the workings. Next to the sandstone walls, the grahamite shows a coarsely granular structure, with a semi-dull fracture. The following layer is highly columnar in structure with a lustrous fracture. Finally, in the center of the vein, the grahamite is more compact and massive, with the columnar structure less developed and a semi-dull fracture. This variation in structure, fracture and lustre is characteristic of grahamite deposits.

On analysis it tests as follows:

(Test 6)	Streak on porcelain	Black
(Test 7)	Specific gravity at 77° F.	1.18–1.185
(Test 9*a*)	Hardness on Moh's scale	2
	Behavior on heating in flame	Softens, burns and splits
(Test 15*a*)	Fusing-point (K. and S. method)	520–540° F.
(Test 19)	Fixed carbon	42.15–42.48 per cent
(Test 21)	Soluble in carbon disulfide	97.61 per cent
	Non-mineral matter insoluble	0.17 per cent
	Combined mineral matter	0.44 per cent
	Free mineral matter	1.71 per cent
	Total	99.93 per cent
(Test 22)	Insoluble in carbon tetrachloride	55.0 per cent
(Test 23)	Soluble in 88° petroleum naphtha	3.0 per cent
(Test 25)	Hydroscopic moisture	0.07 per cent
(Test 26)	Carbon	86.56 per cent
(Test 27)	Hydrogen	8.68 per cent
(Test 28)	Sulfur	1.79 per cent
	Difference	2.97 per cent

Texas.

Fayette and Webb Counties. Clifford Richardson[22] reports a deposit of grahamite in Fayette County in the neighborhood of Lagrange, also an occurrence in Webb County, near Laredo, in the southern portion of the State. These test as follows:

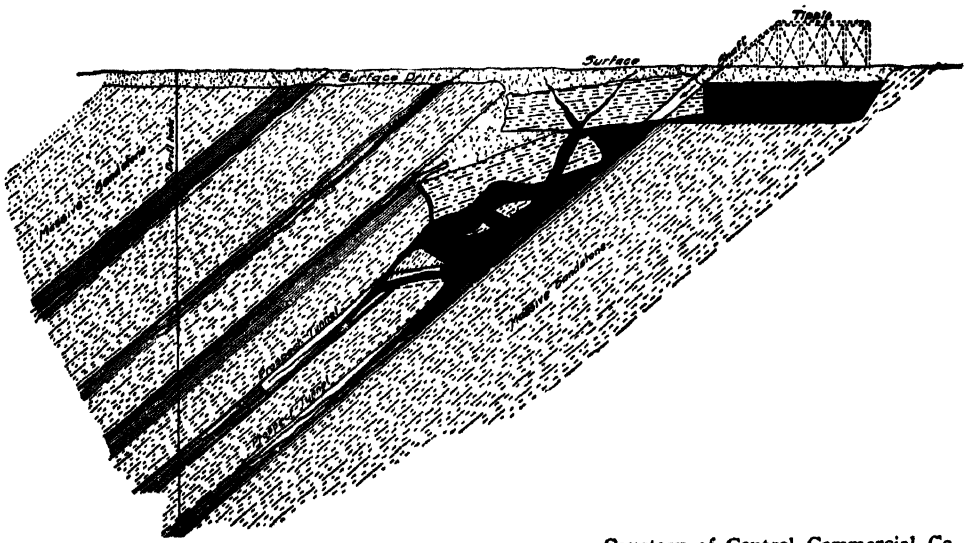
		Fayette County Grahamite, Per Cent.	Webb County Grahamite, Per Cent.
(Test 19)	Fixed carbon	37.7	52.8
(Test 21)	Mineral matter	4.2	2.9
(Test 25)	Moisture	0.3	0.3
(Test 26)	Carbon	76.2	78.6
(Test 27)	Hydrogen	6.6	7.5
(Test 28)	Sulfur	7.4	5.4
(Test 29)	Nitrogen	0.4	1.2
	Undetermined	5.2	5.1

Oklahoma.[23]

Pushmataha County. Two small occurrences are reported in the Potato Hills about 5 miles north of Tuskahoma. One is in SE ¼, Sec. 1, T 2 N R 19 E, and the other in NE ¼, Sec. 2, T 2 N, R 19 E. Neither of these is of importance.

Jackford Creek Deposit. The largest known grahamite vein in the world occurs in Jackfork Valley, 12 miles west of Tuskahoma in

the SE ¼, NE ¼, Sec. 9, T 2 N, R 18 E. It is about 1 mile long, and varies in thickness from 19 to a maximum of 25 ft. At the surface, the vein dips at an angle of 37°, and after continuing downward for 140 ft., turns suddenly at an angle between 45 and 50 deg. It is illustrated in Fig. 84. The grahamite fills a fault in shaly sandstone. The upper wall of the vein is firm and requires no timbering. In mining the material, cave-ins are prevented by allowing pillars of grahamite to remain in place to support the upper "hanging" rock wall. When the author visited the mine in 1912, a track was laid along the bottom wall, and the grahamite hoisted out in skips on



Courtesy of Central Commercial Co.

FIG. 84.—Vertical Section through Grahamite Mine Near Tuskahoma, Okla.

a cable-way. There is evidence of large pieces of rock having become detached from the hanging wall and fallen into the deposit of grahamite before it became solid.

As is common with most grahamite deposits, several distinct types of material are found in the vein. The grahamite which occurs along the rock walls for a thickness of 2 to 6 ft. (type *b*) shows a hackly (known as a "pencillated") fracture, and a semi-dull to dull lustre, whereas the grahamite taken from the center of the vein (type *a*) shows a conchoidal fracture and very bright lustre similar to gilsonite. This is probably due to the fact that the grahamite in contact with the wall cooled more rapidly than the central portion,

and very likely has also been subjected to more or less strain from movements of the surrounding rock. Many thousand tons of grahamite have been mined from this vein which is now pretty nearly exhausted (from 6000 to 7000 carloads during the first four years of its operation, and at the time of the author's visit about 50 tons per day). The cost of moving to the surface is comparatively small, but the material has to be carted 10 miles to Tuskahoma, the nearest shipping point.

On analysis the two varieties (*a*) and (*b*) test as follows:

(Test 1)	Color in mass (types *a* and *b*)	Black
(Test 4)	Fracture (type *a*)	Conchoidal
	Fracture (type *b*)	Hackly
(Test 5)	Lustre (type *a*)	Bright
	Lustre (type *b*)	Semi-bright to dull
(Test 6)	Streak (types *a* and *b*)	Black
(Test 7)	Specific gravity at 77° F. (types *a* and *b*)	1.18–1.195
(Test 9*a*)	Hardness, Moh's scale	2
	Behavior on heating in flame (type *a*)	Intumesces violently
	Behavior on heating in flame (type *b*)	Softens, splits and burns
(Test 15*a*)	Fusing-point (K. and S. method) (types *a* and *b*)	530–604° F.

NOTE.—There is no appreciable difference in fusing-point between the two varieties (*a* and *b*).

(Test 16)	Volatile matter 500° F., 5 hrs.	Less than 1 per cent
(Test 19)	Fixed carbon (types *a* and *b*)	52.76–55.00 per cent
(Test 21)	Solubility in carbon disulfide	Greater than 99.5 per cent
	Non-mineral matter insoluble	Less than 0.5 per cent
	Free mineral matter (types *a* and *b*)	0.21–0.70 per cent

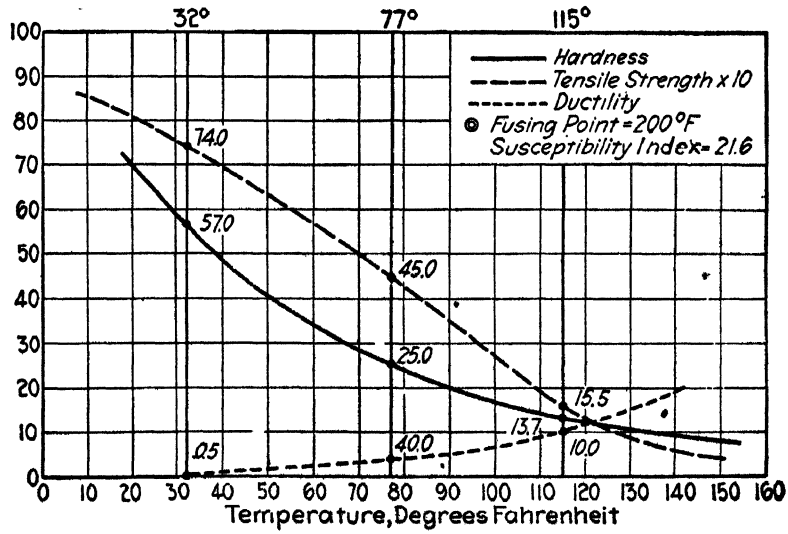


FIG. 85.—Chart of Physical Characteristics of Fluxed Oklahoma Grahamite Mixture.

Figure 85 shows the consistometer hardness, tensile strength (multiplied by 10) and ductility curves of a mixture of the grahamite and residual oil, fluxed together so as to have a hardness of exactly 25.0 at 77° F. (Test 9*c*), equivalent to a penetration at 77° F. of 20 (Test 9*b*). The mixture contains: grahamite, 60 per cent; and residual oil, 40 per cent. The resulting fusing-point (K. and S. method) was 277° F. The same residual oil was used in this test as for the gilsonite in Fig. 76, and the fusing-point of the grahamite used was 550° F. (K. and S. method).[24]

Another mixture containing 33 per cent of the grahamite and 67 per cent of the same residual oil tested as follows:

(Test 9*b*)	Penetration at 115° F., 50 g., 5 sec.	81
	Penetration at 77° F., 100 g., 5 sec.	50
	Penetration at 32° F., 200 g., 60 sec.	55
	Penetration at 0° F., 200 g., 60 sec.	29
(Test 9*c*)	Consistency at 115° F.	5.8
	Consistency at 77° F.	11.7
	Consistency at 32° F.	21.5
	Consistency at 0° F.	37.6
(Test 9*d*)	Susceptibility index	11.7
(Test 10*b*)	Ductility at 115° F.	15
	Ductility at 77° F.	3
	Ductility at 32° F.	1¾
	Ductility at 0° F.	⅛
(Test 15*a*)	Fusing-point (K. and S. method)	135° F.
(Test 15*b*)	Fusing-point (R. and B. method)	147° F.
(Test 16)	Volatile at 500° F., 5 hrs.	0.6 per cent
(Test 17*b*)	Flash-point	535° F.

This particular mixture is characterized by its extremely low susceptibility index and its high ductility, which manifests itself even at 0° F.

Impson Valley Deposit. This occurs on a branch of the Tenmile Creek on the SW ¼, Sec. 21 and NW ¼, Sec. 28, T 1 S., R 14 E., about 16 miles northwest of Antlers. It is known under various names such as Jumbo Mine, Choctaw Mine, or Old Slope Mine. This is the second largest deposit in the State of Oklahoma.

It occurs in a zone of faulting and fracture in shale rock, and the vein is lenticular in form, occurring as a series of pockets of the general form, illustrated in Fig. 32, varying in thickness from a fraction of an inch to 30 ft. as a maximum. As the dip of the vein is very steep, the material must be hoisted out in buckets with a windlass, and then hauled 15 miles to Moyer, the nearest shipping

point. Heavy timbering is necessary on account of the character of the enclosing rock. The grahamite shows the same variation in fracture and lustre as the Jackford Creek deposit. On analysis it tests as follows:

Color in mass, fracture, specific gravity, hardness and behavior on heating in flame, same as the preceding:

(Test 15)	Fusing-point (K. and S. method)	460–520° F.
(Test 16)	Volatile matter, 500° F., 5 hrs.	Less than 1 per cent
(Test 19)	Fixed carbon	48.5–53.0 per cent
(Test 21)	Solubility in carbon disulfide	90.5–96.2 per cent
	Non-mineral matter insoluble	0.0–6.0 per cent
	Free mineral matter	1.1–6.7 per cent
(Test 22)	Carbenes	68 per cent
(Test 23)	Solubility in 88° petroleum naphtha	0.2–0.7 per cent
(Test 25)	Moisture at 100° C.	0.0–0.7 per cent
(Test 26)	Carbon	83.90 per cent
(Test 27)	Hydrogen	7.14 per cent
(Test 28)	Sulfur	1.04–2.24 per cent
	Undetermined	6.72 per cent
(Test 34*a*)	Saturated hydrocarbons	0.32 per cent

Atoka County. McGee Creek Deposits. Two small veins, one 4 in. and another about 1 ft. in thickness, occur in the SW ¼, Sec. 23, T 1 N, R 14 E, about 15 miles northwest of Antlers. These constitute the so-called "William's Mine." Shafts have been sunk from 15 to 20 ft., but not sufficient grahamite has been found to warrant continuing operations. It tests as follows:

(Test 19)	Fixed carbon	43.5–45.7 per cent
(Test 21)	Soluble in carbon disulfide	95.7–99.7 per cent
	Non-mineral matter insoluble	0.0– 4.0 per cent
	Free mineral matter	0.3 per cent
(Test 23)	Soluble in 88° petroleum naphtha	4.5– 6.8 per cent

A larger deposit also occurs in the vicinity of McGee Creek, in the NE ¼, Sec. 25, T 1 S, R 13 E, and NW ¼, Sec. 30, T 1 S, R 14 E, about 12 miles southeast of Stringtown. This is known as the Pumroy or Moulton Mine. The grahamite fills a fissure, caused by faulting, and is reported to be 14 to 15 ft. thick at the surface, tapering to about 4 ft. at a depth of 110 ft. The mine is now abandoned, but when operated some years ago, about 2000 tons were mined annually, being hauled 15 miles to Stringtown, the nearest shipping point. A prospect occurs about ¼ mile south of

the foregoing, consisting of a vein about 2 ft. thick. On analysis it tests as follows:

(Test 15*a*)	Fusing-point (K. and S. method)	473° F.
(Test 19)	Fixed carbon	38.42–41.0 per cent
(Test 21)	Solubility in carbon disulfide	83.7 –95.0 per cent
	Non-mineral matter insoluble	4.8 – 9.2 per cent
	Free mineral matter	0.98– 7.1 per cent

Boggy Creek Deposit. This occurs about 6 miles northeast of Atoka, and 1 mile from the M. K. & T. R. R. in the SW ¼, Sec. 29, T 1 S, R 12 E. The vein occurs in shale varying in thickness from several inches to several feet. It has long been abandoned, and no analyses are available.

Chickasaw Creek Deposit. An undeveloped vein in shale, carrying streaks of grahamite, about 9 ft. thick has been reported in Sec. 15, T 1 S, R 12 E about 2½ miles east of Stringtown on the M. K. & T. Railroad.

Stephens County. This occurs in the NW ¼, Sec. 6, T 2 S, R 4 W, about 6 miles north of Loco, and 18 miles east of Comanche. This vein has been prospected for about half a mile, and occurs as a fault in sandstone and shale. The vein is of a pronounced lenticular type existing in a series of pockets, some as large as 10 ft. across, often connected with a thin vein-like crack less than an inch wide. At several points the deposit pinches out entirely. In the direction of the vein, the pockets measure 25 to 100 ft. horizontally and vertically. A characteristic feature of this deposit is the infiltration of pyrites, grains of which are clearly visible to the naked eye. The surrounding shale is porous, and carries minute particles of the grahamite, which are disseminated throughout the rock for some distance on both sides of the vein.[25]

The material tests as follows:

(Test 4)	Fracture	Hackly
(Test 5)	Lustre	Dull
(Test 6)	Streak	Black
(Test 15*a*)	Fusing-point (K. and S. method)	401–466° F.
(Test 19)	Fixed carbon	34.4 –39.4 per cent
(Test 21)	Soluble in carbon disulfide	81.85–97.70 per cent
	Non-mineral matter insoluble	0.10– 3.60 per cent
	Free mineral matter (mostly pyrites)	2.20–14.55 per cent

Colorado.

Grand County. Deposits of grahamite are found in Middle Park along the continental divide in the northern part of Grand

County. A large vein occurs in Sec. 24, T 4 N, R 77 W, on a fork of Willow Creek about 25 miles north of Grand River, in a region of clay, conglomerate and sandstone. Several veins and fissures have been prospected, the main vein varying in width from 2 in. up to 6 ft., and extending 100 to 125 ft. Comparatively small quantities of the grahamite have been mined, due to difficulties in transportation to the nearest railroad. The product tests:

(Test 7)	Specific gravity at 77° F.	1.15–1.16
(Test 19)	Fixed carbon	47.4 –49.3 per cent
(Test 21)	Soluble in carbon disulfide	98.2 –99.3 per cent
	Non-mineral matter insoluble	0.6 – 1.7 per cent
	Free mineral matter	0.0 – 0.1 per cent
(Test 22)	Carbenes	80.6 per cent
(Test 23)	Solubility in 88° petroleum naphtha	0.8 – 1.3 per cent
(Test 26)	Carbon	85.9 –86.1 per cent
(Test 27)	Hydrogen	7.63– 7.75 per cent
(Test 28)	Sulfur	0.93– 0.99 per cent
	Undetermined	5.34– 5.45 per cent

MEXICO

State of Vera Cruz.

District of Papantla. Grahamite deposits occur about 20 miles south of Papantla, near the village of Espinal, forming a continuation of the glance pitch vein at the village of Talaxca (about 6 miles north of Papantla), previously referred to herein. A total of 5 grahamite veins have been located, with outcroppings at the surface, the widest of which measures 1.5 meters, with an average length of 700 meters. The asphaltite varies from a true grahamite (A) to a metamorphized type (B) approaching impsonite, testing as follows:

		Type "A" (*Grahamite*)	*Type "B"* (*Impsonite*)
(Test 1)	Color in mass	Black	Black
(Test 4)	Fracture	Hackly	Hackly
(Test 5)	Lustre	Bright	Dull
(Test 6)	Streak	Black	Black
(Test 7)	Specific gravity at 77° F.	1.15	1.19
(Test 15*b*)	Fusing-point (R. & B.)	375° F.	Decomposes
(Test 19)	Fixed carbon	37.0 per cent	43.35 per cent
(Test 21)	Soluble in carbon disulfide	99.80 per cent	20.00 per cent
	Non-mineral matter insoluble	0.12 per cent	79.41 per cent
	Mineral matter	0.14 per cent	0.59 per cent
	Total	100.00 per cent	100.00 per cent
(Test 22)	Soluble in carbon tetrachloride	.....	10.3 per cent
(Test 23)	Soluble in 88° petroleum naphtha	39.7 per cent	3.5 per cent
(Test 28)	Sulfur	.....	6.16 per cent

State of San Luis Potosi.

District of Tamazunchale. A vein of grahamite has been found at Huasteca on the Panuco River [26] in a vertical fissure, occurring in shales as an overflow at the junction of the shale stratum with the overlying sandstone. On analysis the material tests as follows:

(Test 4)	Fracture	Semi-conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity, at 77° F	1.145
(Test 19)	Fixed carbon	35.3 per cent
(Test 21)	Soluble in carbon disulfide	93.8 per cent
	Non-mineral matter insoluble	3.4 per cent
	Free mineral matter	2.8 per cent
(Test 23)	Solubility in 88° petroleum naphtha	8.4 per cent
(Test 26)	Carbon	77.67 to 83.14 per cent
(Test 27)	Hydrogen	8.06 to 8.09 per cent
(Test 28)	Sulfur	7.51 to 5.47 per cent

State of Tamaulipas. Another deposit has been reported near the City of Victoria, containing 3.4 per cent of non-mineral matter insoluble in carbon disulfide and 54 per cent of fixed carbon.

CUBA [27]

Province Pinar del Rio. In the District of Mariel, near the City of Bahia Honda, there occurs a fairly large vein of grahamite, known as "La America Mine," or the "Rodas Conception Mine," testing as follows:

(Test 4)	Fracture	Shows distinct cleavage veins
(Test 5)	Lustre	Semi-dull
(Test 7)	Specific gravity, at 77° F	1.157
(Test 19)	Fixed carbon	40.0–42.2 per cent
(Test 21)	Soluble in carbon disulfide	99.4–99.6 per cent
	Non-mineral matter insoluble	0.0– 0.1 per cent
	Free mineral matter	0.4– 0.5 per cent
(Test 22)	Carbenes	About 25 per cent
(Test 23)	Solubility in 88° petroleum naphtha	17.4–20.0 per cent

A smaller deposit, known as "Santa Julia," occurs near the stream of Don Hermanos, 7 miles southwest of Bahia Honda.

Another deposit occurs near the City of Mariel, 1 mile south of Mariel Bay, known as the "Magdalena Mine," which extends about 100 ft. in length and 40 ft. in width. The first concession was granted in 1859 involving 3705 acres. The veins consist of a series of lenses from 2 to over 30 meters wide, in calcareous rock. The

deposit has been worked to a depth of about 300 feet. Large quantities of grahamite of a fairly uniform composition have been mined, characterized by the presence of about 40 per cent of associated mineral matter, which has been utilized principally as a component of paving compositions.[28] It tests as follows:

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.45 to 1.49
(Test 15*b*)	Fusing-point (R. and B. method)	374–400° F.
(Test 16)	Volatile at 212° F	About 5%
(Test 17*b*)	Flash-point (open cup)	415–425° F.
(Test 19)	Fixed carbon	30.0–38.0 per cent
(Test 21)	Soluble in carbon disulfide	51 –58 per cent
	Non-mineral matter insoluble	3 – 7 per cent
	Free mineral matter	38 –42 per cent
(Test 22)	Carbenes	1.5– 6.3 per cent
(Test 23)	Solubility in 88° petroleum naphtha	37 –48 per cent
(Test 26)	Carbon	72.5–77.8 per cent
(Test 27)	Hydrogen	8.5– 8.7 per cent
(Test 28)	Sulfur	6.9– 7.7 per cent
	Difference	6.6–11.4 per cent

Another vein occurs in this same locality, probably a continuation of the preceding, known as the "Mercedes Mine," testing similarly.

Province of Havana. In the neighborhood of Campo Florida, grahamite has been obtained from a mine known as "La Habana," which tests:

(Test 4)	Fracture	Semi-conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.175
(Test 19)	Fixed carbon	45.0 per cent
(Test 21)	Soluble in carbon disulfide	98.9 per cent
	Non-mineral matter insoluble	0.7 per cent
	Free mineral matter	0.4 per cent
(Test 23)	Solubility in 88° petroleum naphtha	6.0 per cent
(Test 26)	Carbon	82.5 per cent
(Test 27)	Hydrogen	7.5 per cent
(Test 28)	Sulfur	6.4 per cent
	Undetermined	3.6 per cent

A similar deposit has been reported about 12 miles east of Havana and another one, known as the "Casitalidad Mine," situated about 9-10 miles east of Havana, and 2 miles south of the coast, in a vein 600-900 ft. long and 1-30 ft. thick, testing substantially the same as the preceding.

Province of Santa Clara. Nine miles northeast of the City of Santa Clara near Loma Cruz, there occurs the deposit known as "Santa Eloisa," in a bed of serpentine. It tests as follows:

(Test 4)	Fracture	Semi-conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.29
(Test 19)	Fixed carbon	34–35 per cent
(Test 21)	Soluble in carbon disulfide	78–79 per cent
	Non-mineral matter insoluble	1.8–2.2 per cent
	Free mineral matter	19–20 per cent
(Test 22)	Carbenes	2–3 per cent
(Test 23)	Solubility in 88° petroleum naphtha	31–33 per cent

Another mine occurs a short distance from Placetas del Sur, in an irregular vein of lenticular form, occurring in several branches. This mine is known as the "Esperanza," and the product is characterized by its comparatively low fusing-point. The average material as mined tests as follows:

(Test 4)	Fracture	Hackly
(Test 5)	Lustre	Moderately bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity, at 77° F	1.22
(Test 9*a*)	Hardness, Moh's scale	2
	In flame	Softens, splits and burns
(Test 15*a*)	Fusing-point (K. and S. method)	400–433° F.
(Test 19)	Fixed carbon	52.95 per cent
(Test 21)	Soluble in carbon disulfide	97.9–98.8 per cent
	Non-mineral matter insoluble	0.05–0.92 per cent
	Free mineral matter	1.15–2.75 per cent
	Mineral matter combined with non-mineral constituents	0.37 per cent

SOUTH AMERICA

TRINIDAD

Two deposits of grahamite [29] occur near San Fernando on the west coast of the island on the shore of the Gulf of Paria, known as the Vistabella and Marbella Mines. The grahamite has been marketed under the name of "manjak," presumably taking advantage of the popularity of the Barbados glance pitch, although from a geological standpoint the two minerals are entirely different. The veins occur in soft shale and sandstone, in a region carrying petroleum in considerable quantities.

A number of veins of grahamite have been uncovered, the largest known as the Vistabella mine, which measures 360 ft. horizontally and has been mined to a depth of about 250 ft. Its thickness is 11 ft. at the outcrop, and increases steadily to 33 ft. at a depth of 200 ft. Three distinct types have been found in the vein, viz:

(1) An amorphous coaly type which has a hackly fracture, and usually occurs at the margin of the vein. It is dull in lustre and exhibits no regular jointings.

(2) A columnar type, of dull lustre, having a columnar jointing running at right angles to the margins of the vein. The jointing is often well formed, dividing the material into hexagonal or pentagonal prisms.

(3) A lustrous variety identical in appearance to gilsonite and Barbados glance pitch (manjak). This has a bright lustre, and a conchoidal fracture, being found in the deeper workings of the mine, at the center of the vein.

There is no chemical difference in the varieties, although it appears that at the center of the vein, at a depth of about 120 ft. the grahamite has a lower fusing-point, closely resembling the Barbados glance pitch, thus serving as a link between the grahamite and the glance pitch, clearly proving that both are derived by metamorphosis from a common source.

A stratum of oil-bearing sandstone is known to exist beneath the grahamite, which appears more than likely to have been derived from an asphaltic petroleum which intruded under pressure through a fault in the shale.

The mining of the grahamite is comparatively simple, but the shafts have to be carefully timbered, and precautions have to be taken to avoid igniting the gases generated in the workings, as these are highly explosive. Formerly between 2000 and 2500 tons were mined per annum.

On analysis it tests as follows:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	3 distinct types recognizable (see above)
(Test 4)	Fracture	Types 1 and 2, hackly; Type 3 conchoidal.
(Test 5)	Lustre	Types 1 and 2 dull; Type 3 bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity, at 77° F	1.170–1.175

(Test 9*a*)	Hardness, Moh's scale	2
(Test 9*b*)	Hardness, penetrometer	0
	On heating in flame	Softens, splits and burns
(Test 15*a*)	Fusing-point (K. and S. method)	350–438° F.

NOTE. The material resembling glance pitch obtained from the centre of the vein at the 200-ft. level fused at 280° F. (K. and S. method).

(Test 15*b*)	Fusing-point (B. and R. method)	370–460° F.
(Test 19)	Fixed carbon	31.5–35.0 per cent
(Test 21)	Solubility in carbon disulfide	91.7–96.0 per cent
	Non-mineral matter insoluble	0.9– 1.2 per cent
	Free mineral matter	4.0– 6.4, averaging about 5.7 per cent
	Mineral matter combined with non-mineral constituents	1.15 per cent
(Test 22)	Carbenes	About 40 per cent
(Test 23)	Solubility in 88° petroleum naphtha:	
	At 100-ft. level	12.8 per cent
	At 140-ft. level	15.2 per cent
	At 200-ft. level	18.5 per cent
	At 200-ft. level, softer material in center	56.0 per cent
(Test 25)	Moisture	0.2–1.0 per cent
(Test 26)	Carbon	84.0 per cent
(Test 27)	Hydrogen	5.7 per cent
(Test 28)	Sulfur	3.0–3.8 per cent
(Test 29)	Nitrogen	2.2 per cent

Figure 86 shows the hardness, tensile strength (multiplied by 10) and ductility curves of a mixture of the grahamite fusing at 400° F (K. and S. method) and residual oil (the same as utilized in mixture shown in Fig 85,) fluxed together in such proportions

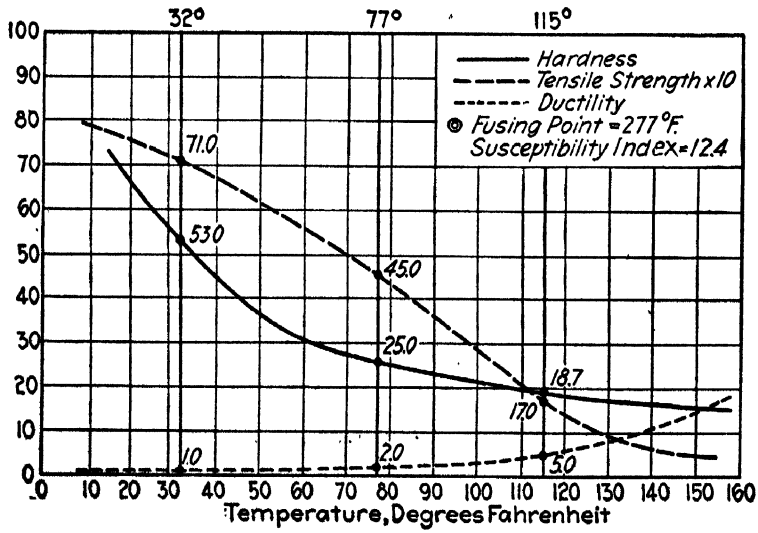


FIG. 86.—Chart of Physical Characteristics of Fluxed Trinidad Grahamite Mixture.

that the hardness at 77° F. is exactly 25.0 (Test 9*c*), corresponding to a penetration at 77° F. of 20 (Test 9*b*). The resulting mixture contained grahamite, 32 per cent and residual oil, 68 per cent, and had a fusing-point of 200° F. (K. and S. method).

The Marbella vein is smaller than the Vistabella, attaining a thickness of 7 ft. near its center. It is lenticular in form and splits up into two smaller veins at one end. The grahamite mined from the Marbella vein has substantially the same characteristics as the preceding. At the 50-foot level, 8.8 per cent is soluble in 88° petroleum naphtha (Test 23); at the 125-ft. level, 9.6 per cent; and at the 200-ft. level 12 per cent.

ARGENTINA

Province of Mendoza. Asphaltite (presumably grahamite?) occurs in several localities, containing up to 0.6 per cent ash carrying 38 per cent vanadium oxide (V_2O_5).[30]

Province of Neuquen. An unusual variety of grahamite occurs on the eastern slope of the Andes Mountains, in the form of a vertical vein about 8 km. long and 2 to 3 m. wide. It must be hauled to the Great Southern Railroad, whence it may be transported to the nearest port, Bahia Blanca. An average sample tests as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.135
(Test 15*a*)	Fusing-point (K. and S. method)	About 625° F.
	Behavior on heating in flame	Decrepitates violently
(Test 21)	Soluble in carbon disulfide	53.35 per cent
	Non-mineral matter insoluble	46.40 per cent
	Mineral matter	0.25 per cent
	Total	100.00 per cent

This asphaltite fluxes with great difficulty with residual oils derived from asphaltic and semi-asphaltic petroleums, which necessitates the mixture being heated to 500° F. for several hours. During this treatment, the "non-mineral matter insoluble" undergoes depolymerization, until it all eventually goes into solution. On the other hand, it fluxes quite readily with linseed oil on heating to 500° F. This grahamite is unusual, in that although half is insoluble in

carbon disulfide, yet it may be fluxed completely as described. It is apparently on the border line between true grahamite and impsonite.

PERU

Province of Tarma.

Department of Junin. Deposits of grahamite occur near Huari, a small town in the Department of Junin, a few miles west of the southern branch of the Central Railway of Peru, at altitudes of 15,000 to 16,000 ft. in the Andes Mountains. They occur as lenticular veins in limestone, and are characterized by the mineral con-

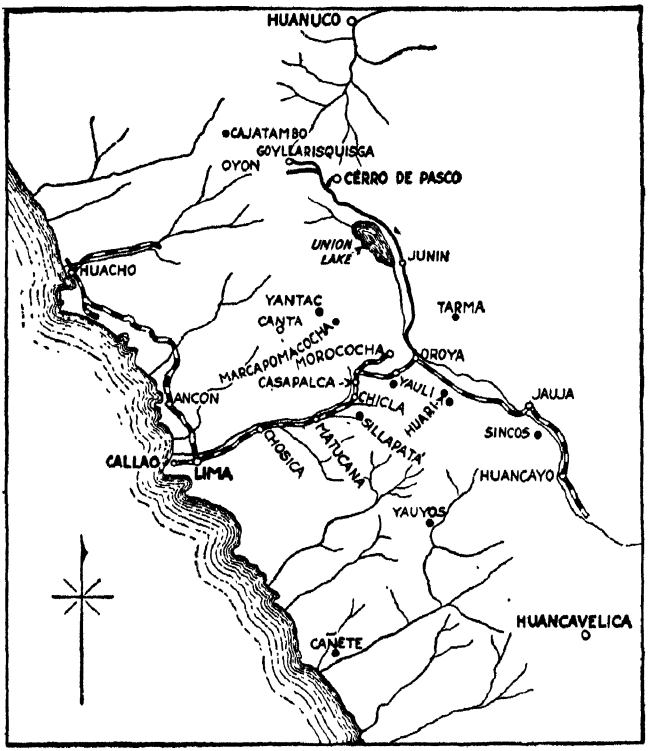


FIG. 87.—Map of Grahamite Region in Peru.

stituents carrying vanadium compounds, probably in the form of sulfide. The region is illustrated in Fig. 87, and includes the following mines: The Chiucho mine consists of a vein 6 in. to 35 ft. wide, 15 miles from the railway, averaging: moisture 2.35 per cent, volatiles 40.45 per cent, fixed carbon 55.0 per cent, and ash 2.20 per cent containing 1.0 to 2.25 per cent vanadic oxide (V_2O_5). La

Lucha mine is connected with the railroad by a small tramway 5 miles long. The output has been utilized extensively as a fuel by the neighboring smelters, and contains: moisture 1.15 per cent, fixed carbon 44.48 per cent, volatiles 48.65 per cent, and ash 5.72 per cent carrying less than 1 per cent vanadic oxide. It is assumed that the vanadium must have been present originally in the asphaltic petroleum from which the grahamite was derived, since it is not found in the associated rocks. In substantiation of this hypothesis, it is pointed out that vanadium is similarly associated (up to 1 per cent) with certain black carbonaceous shales in the province of Jauja, some distance from the foregoing deposits.[31]

Other occurrences have been reported at Minasragra, near Cerro de Pasco, also at Lacsacocha (Yauli), both of which contain vanadium.

CHAPTER XI

ASPHALTIC PYROBITUMENS[1]

The asphaltic pyrobitumens are natural substances composed of hydrocarbons, characterized by their infusibility and comparative freedom from oxygenated substances. They are grouped into five classes, viz.: elaterite, wurtzilite, albertite, impsonite, and asphaltic pyrobituminous shales. The first four are comparatively free from associated mineral matter (usually under 10 per cent). If the mineral matter predominates, the material is known as an asphaltic pyrobituminous shale, which term is applied indiscriminately to shales containing wurtzilite, albertite or impsonite.

Much confusion exists regarding the classification of asphaltic pyrobitumens. Every now and then it is alleged that some new type is discovered, which on closer investigation proves to be an old substance christened under a different name. Thus the so-called "nigrite" described by G. H. Eldridge[2] is nothing more than albertite.

Elaterite, wurtzilite, albertite and impsonite when they occur associated with less than 10 per cent of mineral matter, are distinguished from one another as follows:

	Streak	Specific Gravity at 77° F.	Fixed Carbon, Per Cent
Elaterite	Light brown	0.90–1.05	2–5
Wurtzilite	Light brown	1.05–1.07	5–25
Albertite	Brown to black	1.07–1.10	25–50
Impsonite	Black	1.10–1.25	50–90

All four are derived from the metamorphosis of petroleum, and it is probable that the impsonite represents the final stage of transformation of elaterite, wurtzilite and albertite, as well as the asphaltites (gilsonite, glance pitch and grahamite).

ELATERITE

This asphaltic pyrobitumen is the prototype of wurtzilite. It is found in a few localities, in small amounts and is of scientific interest only.

England

Derbyshire County. Elaterite was originally discovered at the Odin Mine in Castleton by M. Lister in 1673–4.[3] It was again described by C. Hatchett,[4] who found it to be moderately soft and elastic, like India rubber, having a specific gravity of 0.953–0.988. It is slightly soluble in ether (18 per cent) and swells up in petroleum naphtha. H. J. Klaproth [5] examined this same material, stating that it "fuses at a *high heat*, and after this may be drawn into threads between the fingers," also that it contains between 6 and 7 per cent of ash.[6]

Australia

State of South Australia.

Coorong District. A variety of elaterite is found on the coast south of Adelaide, Australia, known under the name of "coorongite." [7] It is a rubbery product, known as "Australian caoutchouc," which shows: fixed carbon 1 per cent, volatiles 97 per cent, and ash 2 per cent. It was deposited on the ground after the subsidence of the floods in 1865 and again in 1920. It is contended that coorongite is derived from algae-like organisms, similar to those which are still to be found in certain salt lakes in South Australia. These organisms appear on the lakes in winter and are blown ashore, where they are supposed to consolidate into coorongite.

Asiatic Russia

State of Turkestan. A deposit occurs at the mouth of the Ili River, in the neighborhood of Lake Balkash,[8] and tests as follows:

(Test 7)	Specific gravity	0.995
(Test 21)	Solubility in carbon disulfide	Very slight
	Free mineral matter	3–5 per cent
(Test 37*a*)	Acid value	4.9
(Test 37*b*)	Saponification value	56.9
(Test 37*c*)	Saponifiable matter	11.1 per cent
	Unsaponifiable matter	88.9 per cent

It is characterized by the presence of saponifiable matter, and in this respect differs from the foregoing. A similar deposit occurs

along Lake Ala-Kool (Ala-Kul), east of the foregoing, in masses 2–10 ft. wide and up to 2 in. thick, associated with algae.[9]

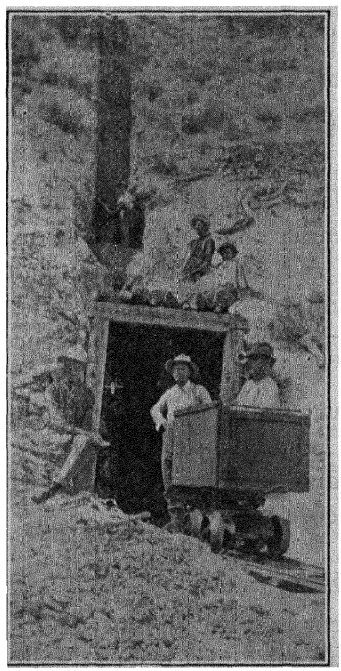
WURTZILITE

This has been found in but one region,[10] as follows:

UNITED STATES

Utah.

Uinta County. This region embraces about 100 square miles in the neighborhood of Indian, Lake, Avintequin and Sams Canyons, tributaries of Strawberry Creek, which in turn leads into the Uinta River. The veins occur about 50 miles southwest of Fort Duchesne, varying in length from several hundred feet to about 3 miles, and from 1 to 22 in. wide, filling vertical faults in shaly limestone. Altogether about 30 veins have been discovered, closely resembling those of gilsonite. Many of them split into a number of smaller branches, either in a vertical or horizontal direction. The largest veins occur between the Left-Hand and the Right-Hand forks of Indian Canyon. Wurtzilite has been exploited under various names, including elaterite (improper use of this name), aegerite, aeonite, etc.

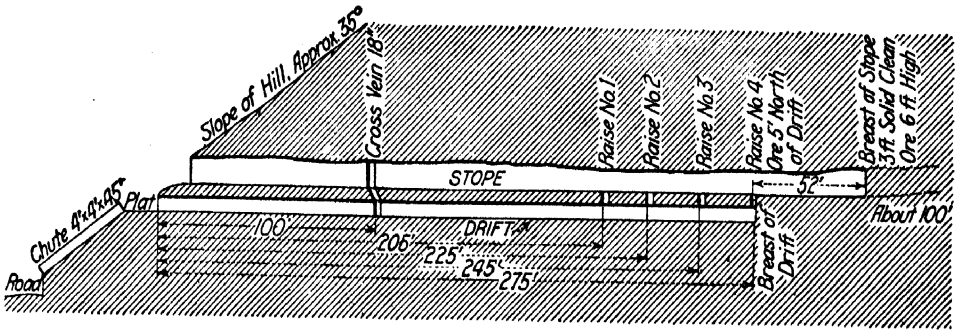


Courtesy of Raven Mining Co.

FIG. 88.—View of Wurtzilite Mine, Uinta County, Utah.

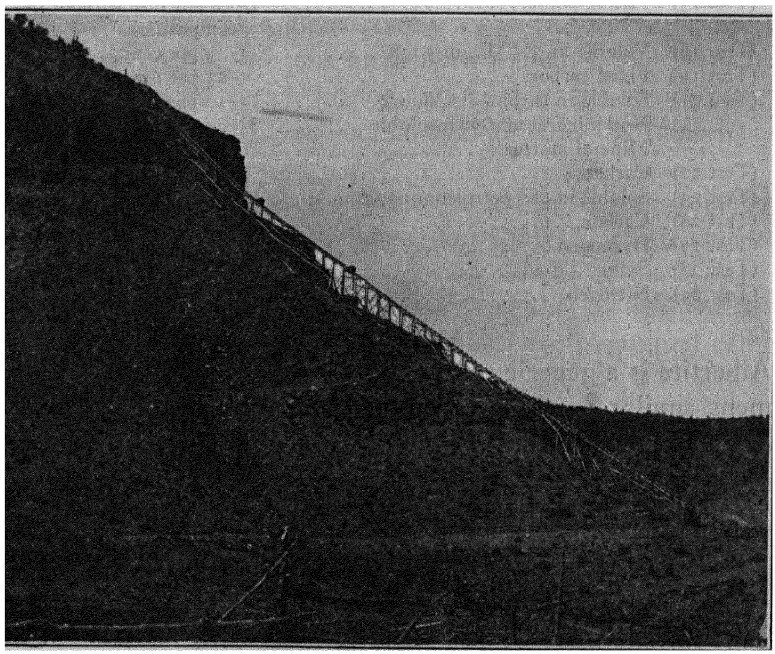
A view of one of the veins is shown in Fig. 88; a section through the mine, in Fig. 89; and the tramway for conveying the product from the hillside mine to the valley below, in Fig. 90.

Wurtzilite is characterized by being sectile and cutting like horn or whalebone. Thin flakes are somewhat elastic, comparable in a way to that of glass or mica, rather than to the yielding elasticity of rubber. If a shaving is bent too far or suddenly, it snaps off like



Courtesy of Raven Mining Co.

FIG. 89.—Vertical Section through Wurtzilite Vein, Uinta County, Utah.



Courtesy of Raven Mining Co.

FIG. 90.—Transporting Wurtzilite from the Mine.

glass. This distinguishes it from other asphaltic pyrobitumens as well as the asphaltites. Attempts were made to find its fusing-point by heating it as high as 800° F. in sulfur, but without having any effect.

It tests as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Light brown
	NOTE. Extremely thin splinters are semi-transparent, showing a deep red color by transmitted light.	
(Test 7)	Specific gravity at 77° F.	1.05–1.07
(Test 9*a*)	Hardness, Moh's scale	Between 2 and 3
(Test 9*b*)	Hardness at 77° F. (penetrometer)	0
(Test 9*c*)	Hardness at 77° F. (consistometer)	Over 150
	On heating in flame	Softens and burns quietly
(Test 15)	Fusing-point	Does not fuse without decomposition
(Test 16)	Volatile at 325° F., in 5 hrs.	1– 3 per cent
(Test 19)	Fixed carbon	5–25 per cent
(Test 21)	Soluble in carbon disulfide	5–10 per cent
	Non-mineral matter insoluble	85–95 per cent
	Mineral matter	0.2–2.5 per cent
(Test 22)	Carbenes	0.0–1.5 per cent
(Test 23)	Soluble in 88° petroleum naphtha	0–2 per cent
(Test 26)	Carbon	79.5–80.0 per cent
(Test 27)	Hydrogen	10.5–12.5 per cent
(Test 28)	Sulfur	4.0–6.0 per cent
(Test 29)	Nitrogen	1.8–2.2 per cent

ALBERTITE

Albertite is a generic term applied to a group of asphaltic pyrobitumens similar to the type-substance which was formerly mined in Albert County, New Brunswick, Canada. The name "carboids" has also been suggested for this group of substances,[11] and the terms "kerotenes" and "kerites" to designate broadly those hydrocarbons that are insoluble in carbon disulfide. Albertites are characterized by their

(1) Infusibility;
(2) Insolubility in carbon disulfide, etc.;
(3) Specific gravity (1.07 to 1.10 at 77° F.);
(4) Percentage of fixed carbon (25 to 50 per cent);
(5) Small percentage of oxygen present in the non-numeral constituents (less than 3 per cent).

It occurs in several localities, of which the typical deposit will be described first.

CANADA

Province of New Brunswick.

Albert County. In 1849 a local geologist, Dr. Abraham Gesner, discovered a substance originally termed "albert coal," subsequently renamed "albertite," [12] on Frederick Brook, a branch of Weldon Creek, near Albert Mines, 20 miles south of Moncton. Shortly after this, litigation gave rise to a discussion whether or not the mineral was a true coal. The courts decided that it was, and not until many years later was its true status determined.

The principal vein has been traced approximately 2800 ft. and varies in thickness from several inches to a maximum of 17 ft. It is connected with a number of smaller lateral veins which in turn break up into still smaller offshoots. The maximum depth reached by mining operations was approximately 1400 ft., and it is estimated that altogether 230,000 tons have been mined. The main use of the product was to enrich bituminous coal in the manufacture of illuminating gas, but it is no longer available, as the mine has been inactive for many years.

This occurrence takes the form of a true fissure vein cutting across a series of beds of so-called "oil shales," which will be described in greater detail later. Mention should be made here that the surrounding shales abound in fossil remains of fish, which indicate that albertite and its associated shales are of animal origin.

On analysis it tests as follows:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	Uniform
(Test 4)	Fracture	Conchoidal to hackly
(Test 5)	Lustre	Bright
(Test 6)	Streak	Brown to black
(Test 7)	Specific gravity at 77° F.	1.075–1.091
(Test 9*a*)	Hardness, Moh's scale	2
(Test 9*b*)	Hardness, penetrometer, 77° F.	0
(Test 9*c*)	Hardness, consistometer, 77° F.	Greater than 150
	On heating in flame	Intumesces
(Test 15)	Fusing-point	Infusible. Decomposes before it melts
(Test 19)	Fixed carbon	25 –50 per cent
(Test 21)	Soluble in carbon disulfide	2 –10 per cent
	Non-mineral matter insoluble	85 –98 per cent
	Mineral matter	0.1–0.2 per cent
(Test 23)	Soluble in 88° petroleum naphtha	0.5–2.0 per cent
(Test 24)	Solubility in pyridine (boiling)	25–35 per cent

		I Per Cent	*II* Per Cent	*III* Per Cent	*IV* Per Cent	*V* Per Cent
(Test 26)	Carbon	83.44	85.40	85.53	86.31	87.25
(Test 27)	Hydrogen	10.08	9.20	13.20	8.96	9.62
(Test 28)	Sulfur	0.44	Trace	1.20	Trace	....
(Test 29)	Nitrogen	....	3.10	0.42	2.90	1.75
(Test 30)	Oxygen	....	2.22	....	1.97	....
	Undetermined	6.04	0.12	....	0.10	1.21
		100.00	100.04	100.35	100.24	99.83

Province of Nova Scotia.

Pictou County. An unusual deposit occurs immediately below the well-known McGregor seam at Stellarton. The approximate thickness of the bed is given as 5 ft., subdivided as follows:

(1) A layer of coal 1 ft. 4 in. wide.
(2) A layer of albertite 1 ft. 10 in. wide.
(3) A layer of pyrobituminous shale 1 ft. 10 in. wide.

This species of albertite has been exploited under the name "stellarite." It seems to represent a state of transition between true albertite and the cannel coals, of which the Scotch mineral torbanite is a representative. The bed contains fossil animal and vegetable remains. A splinter of stellarite may be easily lighted with a match and will burn with a bright, smoky flame, throwing off sparks like stars (whence its name). It was formerly used to enrich bituminous coal in the manufacture of illuminating gas. The layer of coal is an ordinary fat-coking coal, showing a laminated structure, and containing 62.09 per cent of fixed carbon and 4.33 per cent of ash.

The stellarite and associated pyrobituminous shale tests as follows:

		Stellarite (Albertite)	*Pyrobituminous Shale*
(Test 1)	Color in mass	Brown to black	Gray black
(Test 4)	Fracture	Hackly	Conchoidal
(Test 5)	Lustre	Semi-bright to dull	Dull
(Test 6)	Streak	Reddish brown	Brown
(Test 7)	Specific gravity at 77° F.	1.07–1.10	1.56–1.78
(Test 15)	Fusing-point	Infusible	Infusible
(Test 19)	Fixed carbon	22.35–25.23 per cent	8.3–12.3 per cent
(Test 21)	Soluble in carbon disulfide	2.0 per cent	Trace
	Mineral matter	8.2–8.9 per cent	52.0 –62.0 per cent
(Test 25)	Moisture	0.2–0.3 per cent	0.6 – 1.0 per cent
(Test 26)	Carbon	88.1 per cent	.....
(Test 27)	Hydrogen	11.1 per cent	.....
(Test 28)	Sulfur	0.1 per cent	0.25– 0.74 per cent
(Test 29)	Nitrogen	0.2 per cent	.....
(Test 30)	Oxygen	0.5 per cent	.....

The presence of the very small percentage of oxygen (0.5 per cent) differentiates the material from lignite and the other non-asphaltic pyrobitumens, thus corresponding with the ultimate analysis of the New Brunswick albertite.

UNITED STATES

Utah.

Uinta County. A vein of albertite (christened "nigrite" by Eldridge), 120 ft. long, showing a maximum width of 20 in., is found 8 miles from Helper, and 5 miles east of Soldier Summit, having the following characteristics:

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Semi-dull
(Test 6)	Streak	Brownish black to black
(Test 7)	Specific gravity at 77° F.	1.092–1.099
(Test 9*a*)	Hardness, Moh's scale	2
	Behavior on heating in flame	Splits and burns
(Test 15)	Fusing-point	Infusible
(Test 19)	Fixed carbon	37–40 per cent
(Test 21)	Soluble in carbon disulfide	3– 6 per cent
	Non-mineral matter insoluble	94.2–97.0 per cent
	Mineral matter	0.2 per cent
(Test 23)	Soluble in 88° petroleum naphtha	Trace
(Test 28)	Sulfur	1.0 per cent

SOUTH AMERICA

Falkland Islands.

An asphaltite resembling albertite, having a specific gravity at 77° F. of 1.04, containing 3.7 per cent of siliceous ash, and mostly insoluble in the usual solvents, has been reported.[13]

GERMANY

Province of Hanover. A small deposit of albertite-like pyrobitumen was discovered in 1870 south of the village of Bentheim, east of the city of Gildehaus. The material was mined years ago and utilized as fuel, under the name "Gagat-kohle." It occurs in a fault of a deposit of clayey schist, in a vein 0.50 to 0.65 m. wide. The material has a high lustre, a conchoidal fracture, a black streak, specific gravity at 77° F. 1.075, hardness 2.5, soluble in carbon disulfide 23.5 per cent, slightly soluble in turpentine, ash 0.53 per cent. It softens and swells in a flame and distills without fusing. The mine is now idle.[14]

AUSTRALIA

Tasmania. A species of albertite described under the name of "tasmanite" [15] has been reported near the River Mersey in the northern portion of Tasmania. It is found disseminated in a pyrobituminous shale and complies with the following tests:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	Uniform
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Yellowish brown
(Test 7)	Specific gravity at 77° F	1.10
(Test 9*a*)	Hardness, Moh's scale	2
(Test 15)	Fusing-point	Infusible
(Test 21)	Soluble in carbon disulfide	Trace
	Mineral matter	8–14 per cent
(Test 26)	Carbon	79.2 –79.3 per cent
(Test 27)	Hydrogen	7.2 – 7.4 per cent
(Test 28)	Sulfur	5.28– 5.32 per cent
(Tests 29 and 30)	Nitrogen and oxygen	4.93 per cent

PORTUGUESE WEST AFRICA

Province of Angola.

District of Libollo. A species of albertite is reported at Calucala, 14 miles north of the railway station Zenza do Itombe (which is 18 miles east of Luanda on the Luanda-Malange Railway line) under the name "libollite." [16]

IMPSONITE

This represents the final stage in the metamorphosis of asphaltites and asphaltic pyrobitumens. It is characterized by its:

(1) Infusibility and insolubility in carbon disulfide;
(2) Specific gravity (1.10 to 1.25);
(3) High percentage of fixed carbon (50 to 85 per cent);
(4) Comparatively small percentage of oxygen (less than 5 per cent), which differentiates it from the non-asphaltic pyrobitumens.

The weathered asphaltites taken from the exposed portions of the vein, where they have been subjected for centuries to the action

of the elements, closely resemble impsonite in their physical and chemical properties, and may therefore be classified as such. Outcrops of grahamite are especially prone to metamorphize into impsonite, and many prospectors have been misled on this account.

The following represent the most important deposits:[17]

NORTH AMERICA

UNITED STATES

Oklahoma.

La Flore County. One of the largest deposits of impsonite occurs 2 miles east of Page on the southern slope of Black Fork Mountain (S ½, Sec. 24, T 3 N, R 26 E), filling a fissure caused by a fault. The vein is about 10 ft. thick, and has been mined to some depth. It complies with the following tests:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Hackly
(Test 5)	Lustre	Semi-dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.235
(Test 9*a*)	Hardness, Moh's scale	2–3
	Heating in flame	Decrepitates
(Test 15)	Fusing-point	Infusible
(Test 19)	Fixed carbon	75.0–81.6 per cent
(Test 21)	Soluble in carbon disulfide	4– 6 per cent
	Non-mineral matter insoluble	93–96 per cent
	Mineral matter	0.7–2.5 per cent
(Test 24)	Solubility in pyridine (boiling)	3.88 per cent
(Test 25)	Moisture	0.1–1.5 per cent
(Test 28)	Sulfur	1.69 per cent

Murray County. Impsonite has been reported 5 miles northeast of Dougherty (Sec. 33, T 1 S, R 3 E), in a vein about 18 in. thick at the top and 7 ft. at the bottom. Its characteristics are similar to the preceding.

Arkansas.

Scott County. Another deposit of impsonite, referred to as "arkosite,"[18] occurs in the western part of Fourche Mountain, about 12 miles east of the Black Fork Mountain locality in Oklahoma. The exact locality is 1 mile east of Eagle Gap, and 2 miles

east of Harris. It occurs in a region of shale and sandstone, and tests as follows:

(Test 1)	Color in mass	Black
(Test 4)	Fracture	Hackly
(Test 5)	Lustre	Semi-dull
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F	1.25
(Test 9*a*)	Hardness, Moh's scale	3
	Heating in flame	Decrepitates
(Test 15)	Fusing-point	Infusible
(Test 19)	Fixed carbon	80.0 per cent
(Test 21)	Soluble in carbon disulfide	Trace
	Non-mineral matter insoluble	99.3 per cent
	Mineral matter	0.6 per cent
(Test 28)	Sulfur	1.38 per cent

Nevada.

Eureka County. A deposit is reported 15 miles south of Palisade in Pine Creek valley, in a vein filling a fault about 300 ft. long and of unknown depth. Its physical and chemical characteristics are similar to the preceding.[19]

Michigan.

Keweenaw County. An interesting formation has been described in the vicinity of the Porcupine Mountains in the southwest part of Keweenaw Point, in which the impsonite acts as a cement in sandstone beds carrying grains of native copper. Much of the impsonite is surrounded by copper, giving rise to the assumption that the former was present prior to the deposition of the copper. The impsonite analyzes: 64.8 per cent fixed carbon and 33.3 per cent ash containing 2 per cent copper.[20]

SOUTH AMERICA

PERU [21]

Provinces of Canta (Department of Lima) and Yauli (Department of Junin). More than a dozen veins have been found in the District of Yantac, about 28 miles from Casapalica, on the Central Railway of Peru, near the city of Marcapomacocha (see Fig. 87), also at Cajatambo (Coxitambo), having a specific gravity of 1.25, a

bright conchoidal fracture, a black streak and 1.6 per cent ash.[22] They occur along both slopes of the western Cordillera of the Andes, at altitudes of 12,000 to 16,000 ft., and vary in thickness from a few inches to 5 ft. The average composition shows: moisture 5.2 per cent, fixed carbon 72.2 per cent, volatile 10.3 per cent, and ash 12.3 per cent (containing 5 to 16 per cent of vanadic oxides V_2O_5). The presence of tiny fissures radiating from the main vein, and the inclusion of "horses" in the vein proper differentiate the material from a coal formation. It is, however, distinctly anthracite in character, burning with little to no flame.

Llacsacocha Mine occurs 13 miles by trail south of the town of Yauli, extending for more than 1200 ft. in limestone formation, having numerous lenses and faults. It is black and lustrous, free from pyrites, and contains: fixed carbon 88.2 per cent, volatile 9.0 per cent, and ash 2.8 per cent (carrying 15 per cent vanadic oxide).

Rumichaca Mine at Minasragra, near Cerro de Pasco, yields about 80 per cent of the world's supply of vanadium and is the largest of all in the Yauli district. Being close to the railroad, it has been largely worked during the past ten years, and used as a fuel. The vein carries from a few inches to 40 ft. in width and contains an average of 16 per cent ash (carrying 5.3 per cent vanadic oxide)—otherwise it is similar in composition to the foregoing.

Negrita Mine is situated 15 miles from the railroad, near Hullacocha Lake, occurring as a true fissure vein, cutting across limestone and shale. Three lenses yielded 2000 tons. An average analysis showed 9.5 per cent ash (carrying 5 per cent vanadic oxide).

Cacharata Mine is located about 1000 yd. from the previous vein and measures 2 ft. wide, being similar in composition.

Province of Huarochiri (Department of Lima). Narrow and irregular veins occur at Sillapata, 15 miles from the railway station of Matucana, running high in ash (averaging slightly less than 1 per cent of vanadic oxide).

Brazil

State of São Paulo. Impsonite has been found in a dyke near Limeira, analyzing: fixed carbon 76 per cent, volatile matter 5 per cent, and ash 16 per cent.[23]

AUSTRALIA

West Australia. A deposit of impsonite has been reported in West Australia having a specific gravity of 1.154, moisture 0.37 per cent, fixed carbon 56.27 per cent, volatiles 41.91 per cent, and ash 1.82. It does not melt up to 300° C.[24]

CHAPTER XII

PYROBITUMINOUS SHALES[1]

Under this heading will be considered the *oil-forming* shales containing pyrobitumens associated with earthy matter, which will produce oily or tarry distillates upon being subjected to destructive distillation. *Oil-bearing* and *asphalt-bearing* shales from which petroleum or asphalts may be extracted with solvents are not included. The well-known shales in France occurring at Autun (Saône-et-Loire) and Bruxières-les-Mines (Allier) consisting of semi-liquid asphalt associated with shales shall accordingly be excluded, although these have been classified indiscriminately with the true pyrobituminous shales by other writers.

Pyrobituminous shales may be sub-divided into two classes:

(1) Asphaltic pyrobituminous shales in which asphaltic pyrobitumens (elaterite, wurtzilite, albertite or impsonite) are associated with shales.

(2) Non-asphaltic pyrobituminous shales in which non-asphaltic pyrobitumens (cannel coal, lignite or bituminous coal) are associated with shales.

Little or no attempt has been made to differentiate between these two groups, on account of the difficulty in identifying the bituminous material present. This will become apparent when it is considered that pyrobitumens are more or less insoluble in solvents and are moreover masked by the associated mineral matter, which interferes with the usual distinguishing tests, such as the specific gravity, lustre, streak, etc. Up to the present time all pyrobituminous shales have been referred to under the general term "oil shales," which is really a misnomer.

The following means are suggested to differentiate the two classes:

(1) By the pyrobitumens found locally.

The presence of asphaltic bitumens in the vicinity would indicate an asphaltic pyrobituminous shale. Similarly, non-asphaltic pyro-

bitumens would tend to establish the identity of the shale as non-asphaltic. If both types are present, the evidence is non-conclusive.

(2) By the associated fossil remains.

If vegetable (plant) fossil remains *only* are found associated with the shale, the indications are that it is non-asphaltic, since it is definitely established that the non-asphaltic pyrobitumens are of *vegetable* origin. On the other hand, if animal (fish or mollusc) fossil remains are present, the shale will more than likely represent the asphaltic pyrobituminous variety.

(3) Effect of heat on the solubility.

On heating in a closed retort to 300° to 400° C., asphaltic pyrobituminous shales will depolymerize and become more soluble in carbon disulfide, whereas the non-asphaltic pyrobituminous shales remain unaffected. It is also interesting to note that the bituminous constituents of non-asphaltic pyrobituminous shales are rendered fusible and soluble by heating the finely powdered shale to 500 to 600° F. in a closed retort with a residual oil derived from petroleum, or with coal tar.[2]

(4) By the percentages of fixed carbon and oxygen (calculated on the basis of the non-mineral matter present). These two criteria, considered together, furnish the most reliable means of distinguishing between the two classes of shale, as will be observed from the following figures, calculated on the basis of the non-mineral constituents present:

	Fixed Carbon (Calculated on the Ash-free Basis) Per Cent	Oxygen (Calculated on the Ash-free Basis) Per Cent
Asphaltic Pyrobituminous Shales:		
Wurtzilite shales	2–10	Less than 2
Albertite shales	5–25	Less than 3
Non-asphaltic Pyrobituminous Shales:		
Cannel coal shales	5–20	5–10
Lignite shales	15–30	15–28
Bituminous coal shales	25–50	3–18

It will be noted that the percentage of fixed carbon calculated on the mineral-free basis, runs lower than in the corresponding (pure) pyrobitumens, due to the presence of the mineral matter, which assists in the combustion of the carbon during the test, decreasing the yield of "fixed carbon," and at the same time increasing the percentage of volatile constituents. This is important from a com-

mercial viewpoint. The most valuable pyrobituminous shales are those which produce the largest amount of volatile matter when subjected to destructive distillation. This is true with the albertitic, cannel coal (torbanitic) and lignitic shales, whereas the bituminous coal shales yield but little volatile matter and have no commercial importance.

Two types of bituminous constituents are present in non-asphaltic pyrobituminous shales, viz.: (1) macerated and carbonized plant remains similar to coal, and (2) yellow resinous bodies representing the last stage in the oxidation of the woody tissue. Elaterite, wurtzilite and impsonite shales are rarely found.

Pyrobituminous shales generally contain more than 33 per cent of associated mineral constituents. The non-mineral constituents present in non-asphaltic pyrobituminous shales have been designated by the terms "kerogen" [3] or "petrologen." [4]

From the foregoing it will be apparent that the subject of "pyrobituminous shales" is an extremely complicated one, still requiring a vast amount of research work before all the deposits can be correctly classified.

Pyrobituminous shales are treated exclusively by subjecting them to a process of destructive distillation in suitable retorts to recover the tarry distillate and ammonium sulfate as will be described in Chapter XVI. The intrinsic value of the shale is dependent upon the amount of shale tar and ammonium sulfate obtained.

A detailed description of the individual deposits of pyrobituminous shales does not fall within the scope of this publication.

PART III

TARS, PITCHES, AND PYROGENOUS ASPHALTS

CHAPTER XIII

GENERAL METHODS OF PRODUCING TARS

Tars constitute the volatile oily decomposition products obtained in the pyrogenous treatment of bituminous and other organic substances. The pyrogenous treatment embraces three processes, viz.:

(1) Subjecting to heat alone without access of air, often termed "destructive distillation," and sometimes referred to as "pyrolysis."

(2) Partial combustion, which may take place either in an atmosphere of air and steam (in gas producers) or with a limited access of air.

(3) Cracking oil vapors at high temperatures.

Practically all organic substances which undergo decomposition produce tars upon being subjected to heat, provided they yield a substantial proportion of volatile decomposition products, the temperature is sufficiently high to bring about the decomposition, and air is entirely or partially excluded during the pyrogenous treatment. If the organic substance does not contain volatile matter, as proves the case with anthracite coal or graphite, no tar will result. If air is present in too large a quantity, the products of decomposition will undergo *complete* combustion, and the tar will be consumed. Materials which evaporate (*i. e.*, distil undecomposed), or sublime, will remain unchanged in composition, and products which explode are converted into permanent gases, without the formation of tars.

At the present time tars are produced commercially from the following products:

(1) Bituminous substances including peat, lignite, bituminous coal, petroleum and pyrobituminous shales.

(2) Certain other organic substances including wood, and bones.

The following will give a synoptical outline of the raw materials used, the modes of treatment and the kinds of tar produced:

Raw Materials Used	Heat Alone ("Destructive Distillation")	Partial Combustion		"Cracking"
		Air and Steam ("Producers")	Limited Access of Air	
Bituminous substances:				
Petroleum products	Wax tailings	.....	.....	Pressure tar; Oil-gas tar; Water-gas tar
Peat	Peat tar	Peat tar	.....	.....
Lignite	Lignite tar	Lignite tar	.....	.....
Pyrobituminous shales	Shale tar	Shale tar	.....	.....
Bituminous coals	Gas-works coal tar; Coke-oven coal tar	Producer-gas coal tar	Blast furnace coal tar	.....
Other Organic Materials:				
Wood	Wood tar	.....	.....	.....
Bones	Bone tar	.....	.....	.....

Petroleum products (e.g., "gas oils") upon being subjected to a high temperature under more or less pressure in a closed retort will result in the formation of oil-gas tar; and when sprayed on incandescent anthracite coal or coke result in the production of water-gas tar. Peat, lignite and pyrobituminous shales result in the formation of peat-, lignite- and shale-tars respectively: (1) when subjected to destructive distillation, or (2) upon undergoing partial combustion in an atmosphere of air and steam in a so-called "gas producer." Tars resulting from these two processes are similar in composition and hence are designated by the same name. Destructive distillation yields a larger percentage of tar than partial combustion in an atmosphere of air and steam.

Bituminous coals form different kinds of tar, depending upon the nature of the process. Thus gas-works coal tar and coke-oven coal tar are produced by the destructive distillation of bituminous coal in gas-works retorts and coke-ovens respectively. Producer-gas

coal tar is derived from the partial combustion of bituminous coal in an atmosphere of air and steam in a gas producer. Blast-furnace coal tar results from the partial combustion of bituminous coal in a limited access of air in a so-called "blast furnace." Destructive distillation of wood results in the formation of wood tar, and of bones in the production of bone tar.

In the order of their commercial importance, based on the quantities produced annually, tars may be grouped as follows, viz.: coal tar is produced in the largest quantity, water and oil-gas tars come next, and wood tar follows in sequence. Insignificant quantities of producer-gas coal tar, bone tar, blast-furnace tar, peat-, lignite- and shale-tars are produced in the United States. Lignite tar is produced in comparatively large quantities in Germany. The production of peat- and bone-tar has not assumed great importance anywhere.

The following tars have been described in the literature,[1] produced by the destructive distillation of the corresponding substance, but have but little commercial value:

Montan-wax tar;[2] beeswax-tar; tars derived from the destructive distillation of vegetable and animal oils; linoleum tar derived from linoleum waste; cork tar; leather tar;[3] tanning residues; seaweed tar; sulfite cellulose tar (tall oil tar) produced from the destructive distillation of sulfite cellulose liquor;[4] lignin tar; amber tar, also tars derived from other fossil resins; straw tar; tars derived from seed husks and hulls;[5] tobacco tar; bagasse tar;[6] molasses tar;[7] tar from beet residues in sugar manufacture; tar from potato residues resulting from fermentation;[8] tars from fermentation residues of various sorts; vinasse tar; anthracene-oil tar obtained as residue in anthracene oil distillation; fusel-oil tar obtained on distillation of fusel oil; asphalt tar obtained on destructive distillation of natural rock asphalts (e.g., at Ragusa, Italy; Tyrol, Austria; and at other localities), etc.

The corresponding pitches are produced by distilling the respective tars listed above; i.e., montan-tar pitch, beeswax-tar pitch, vegetable- (or animal) oil-tar pitch, linoleum-tar pitch, cork-tar pitch, leather-tar pitch, tannin-tar pitch, seaweed-tar pitch, sulfite-cellulose-tar pitch (tall-oil pitch), lignin-tar pitch, amber-tar pitch, straw-tar pitch, seed-tar pitch, tobacco-tar pitch, bagasse-tar pitch, molasses-tar pitch, beet-residue-tar pitch, potato-residue-tar pitch, fermenta-

tion-residue-tar pitch, vinasse-tar pitch, anthracene-oil-tar pitch, fusel-oil-tar pitch, naphthylamine pitch, cumarone pitch, cresol pitch (carbol pitch), asphalt-tar pitch, etc.

We will now consider the various processes for producing tars in greater detail.

DESTRUCTIVE DISTILLATION

This process is used for destructively distilling *infusible* organic substances including non-asphaltic pyrobitumens, pyrobituminous shales, wood and bones. It consists in heating the substance to a high temperature in a still from which air is excluded, and the distillation is continued until the volatile constituents are driven off and the residue carbonizes. The volatile constituents are grouped into two classes, viz.: non-condensable and condensable products, the former including the permanent gases, and the latter, the aqueous liquor and tar.

The nature of the ingredients formed during the distillation depends largely upon the nature of raw material used and the temperature at which it undergoes decomposition. As a rule, the older the substance from a geological standpoint, the higher will be the temperature at which it decomposes. At low temperatures, we find aliphatic (straight chain) hydrocarbons in the tar, also varying amounts of phenolic bodies, of toluene and naphthalene, but no benzene or anthracene. This is true in the case of peat, lignite, cannel coal and pyrobituminous shales. Where the destructive distillation takes place at a high temperature, aromatic hydrocarbons will predominate, including benzene and anthracene. This is true with bituminous coals. The aqueous liquor will show an acid reaction in the case of wood and peat, and an alkaline reaction with lignite, coals and pyrobituminous shales.

In general, the yield of tar depends upon five factors, viz.: the composition of the substance, the temperature, the time of heating, the pressure, and upon the efficiency of the condensing system. These will be considered in greater detail.

The Composition of the Substance. (*a*) *The Percentage of Volatile Constituents.* The greater the percentage of volatile constituents, and conversely the smaller the percentage of "fixed carbon," the larger will be the yield of tar. Figured on the basis of

the dry weight of the non-mineral constituents, the yield of volatile matter will range as follows, commencing with the highest: wood, peat, lignite, bituminous coal. The yields of tar follow in the same sequence, viz.:

Wood	10 –20 per cent
Peat	7½–15 per cent
Lignite	5 –10 per cent
Bituminous coal	3 – 7 per cent

(*b*) *The Percentage of Oxygen in the Fuel.* As a general rule, the greater the percentage of oxygen in the fuel, the greater will be the yield of tar. Georg Lunge [9] cites the following figures to show the relation between the percentages of oxygen, tar, and water, based on the dry weight of fuel:

Fuel Contains Per Cent	Yield Tar, Per Cent	Yield Water, Per Cent
Oxygen 5–6½	3.30	4.58
Oxygen 6½–7½	4.65	5.86
Oxygen 7½–9	5.08	6.80
Oxygen 9–11	5.48	8.60
Oxygen 11–13	5.59	7.86

The Temperature. (*a*) *The Temperature at which the Fuel Decomposes.* As stated previously, each type of fuel has a definite temperature at which distillation commences. The older the fuel from a geological standpoint, the higher will be this temperature, and hence the greater will be the yield of coke, and the smaller that of tar. It would appear that a preliminary decomposition approaching a state of fusion occurs at this temperature, which remains fairly constant until the carbonization is complete. The coke-forming property of bituminous coals depends upon the presence of constituents melting at a lower temperature than that at which carbonization occurs.

(*b*) *The Temperature at Which the Distillation Is Performed.* This is distinct from the preceding, and is determined by the quantity and intensity of the heat applied externally to the retort in which the destructive distillation takes place. It depends upon the nature of the heating medium, and the manner in which it is applied. The temperature may be close to that at which the fuel undergoes

distillation, or it may be vastly in excess thereof. The higher the temperature above that necessary to cause incipient decomposition, the smaller will be the yield of tar, and the larger that of gas; moreover, a high temperature results in the formation of a larger percentage of free carbon in the tar, due to greater decomposition ("cracking") of the distillate.

Aromatic hydrocarbons, upon being subjected to a gradually increasing temperature (650–800° C.), are transformed as follows:

Higher benzene homologues→Lower benzene homologues→Diphenyl→Naphthalene→Anthracene.

At temperatures above 800° C., the anthracene is decomposed into carbon and gas.[10]

The Time of Heating. (*a*) *Thickness of the Fuel Layer.* The deeper the layer of fuel in the retort or furnace, the greater the super-heating, and consequently the smaller will be the yield of tar and the larger that of gas. When the layer is deep, the volatile portions are compelled to pass through a mass of incandescent fuel, so that the temperature of the gases is increased, due to the greater *time* of contact. This is the underlying principle in the manufacture of generator gas.

It follows also that the greater the area of contact between the fuel and the heating surface, the shorter time it will take to raise the temperature of the former the requisite degree. Small charges of fuel may thus be heated more rapidly, which is conducive to the formation of a greater proportion of gas and tar and a smaller yield of coke. Slow heating, on the other hand, results in the production of a large proportion of coke, and smaller proportions of gas and tar respectively. It is for this reason that comparatively small and narrow retorts are used for the manufacture of illuminating gas, and very much larger chambers where coal is treated to obtain coke.

(*b*) *Size of the Fuel.* The size of the lumps of fuel has an important bearing on the time of heating. If the lumps are too fine, they will pack together to such an extent that insufficient space is left between them for the transfer of heat by the gaseous products. On the other hand, if the lumps are too large, it will take an abnormally long time for the carbonization process to reach the center of each lump, since the heat conduction of the fuel itself is poor.

(*c*) *Construction of the Retort or Furnace.* The thickness of the walls, the method of heating, the size as well as the nature of the material of which the retort is constructed, all tend to influence the time of heating. Small units, the use of preheated gases for supporting the combustion, and thin retort walls constructed of materials which have a relatively high conductivity at elevated temperatures, serve to decrease the time of heating.

The Pressure. The greater the pressure, the longer are the volatile products forced to remain in contact with the hot retort and incandescent fuel, and the greater, therefore, will be the carbonization. The use of reduced pressure hastens the removal of the volatile constituents and serves to increase the outputs of gas and tar, and reduce the yield of coke. At the same time, the period of distillation is increased. In manufacturing illuminating or fuel gas, modern practice consists in carrying out the distillation under a moderate vacuum. On the other hand, when the main object is to produce coke, the pressure of the gas inside the retort is purposely allowed to increase somewhat.

The Efficiency of the Condensing System. As the vapors leave the retort, oven, blast-furnace, or producer at 500 to 800° C., all the constituents exist in the gaseous state, excepting the "free carbon" derived from the decomposition of the gases in contact with the highly heated walls, and the particles of mineral matter which are carried over mechanically. The vapors are composed of a mixture of substances, some congealing to solids, others condensing to liquids, and still others remaining as permanent gases at atmospheric temperature and pressure. As the vapors cool, the solids and liquids separate out, forming the tar. This separation is progressive, the higher boiling-point constituents condensing first, followed by substances of lower boiling-points, and finally liquids boiling slightly above atmospheric temperature. With this in view, the vapors may either be cooled slowly, or they may be cooled rapidly, so that all condensable constituents are caught together in the form of "tar," to be redistilled later into its components. It is a singular fact, that even when the vapors have been thoroughly cooled, the tar will not separate out completely, without further treatment. Part remains suspended in the gases as infinitesimally fine globules, known as a "tar fog." This term is most expressive, since its behavior is very

similar to that of an ordinary fog, alluding to the weather. Mere cooling will not condense a "tar fog," accordingly other means must be employed.

PARTIAL COMBUSTION WITH AIR AND STEAM

This takes place in manufacturing producer gas. Several forms of producers are in use, and peat, lignite, pyrobituminous shale, or bituminous coal are variously employed as fuel. The reaction which ensues may be expressed as follows, in which "C" represents the carbonaceous matter present in the form of fuel:

$$2C + O + H_2O = 2CO + H_2$$

The resulting gas, known as "producer gas," is composed of carbon monoxide with a smaller proportion of hydrogen. When anthracite coal or coke is used as fuel, no tar results; with bituminous coal, tar is formed in certain types of producers but not in others; and with peat or lignite, tar is produced in all types, on account of the readiness with which they volatilize at low temperatures, and the comparatively large proportion of volatile constituents present. These tars correspond very closely in physical and chemical properties to the ones obtained from the corresponding processes of destructive distillation, but with the former the yield is smaller, since most of the tarry matter is consumed.

Lignite carrying a *moderate* proportion of mineral matter (*e.g.*, Messel lignite) is treated in a special form of producer to obtain a small amount of gas and the largest possible yield of tar (4 to 14 per cent). This is brought about by introducing a *limited* and carefully regulated quantity of air and steam, sufficient only to support partial combustion. The same method is always followed in treating pyrobituminous shales, on account of the greater intrinsic value of the tar, of which 5 to 25 per cent is recovered. These processes approach destructive distillation closely, the object being to bring about incipient combustion of the lignite or shale and the non-condensable gases derived therefrom, thereby raising the temperature sufficiently to cause destructive distillation.

When peat, bituminous coal or lignite containing a *large* proportion of mineral matter is treated in a producer, it is always intended

to produce the largest possible yield of gas, and the smallest proportion of tar.

PARTIAL COMBUSTION WITH A LIMITED ACCESS OF AIR

This process takes place in manufacturing generator gas, also upon smelting ores in blast-furnaces. No tar is produced in manufacturing generator gas, hence this process ceases to be of interest from the bituminologist's standpoint. In the case of blast-furnaces, no tar results when anthracite coal or coke is used as fuel, but when bituminous coal is used, as is sometimes the practice in England and on the Continent, 2 to 3½ per cent of its weight of tar is produced. The air is forced into the blast-furnace from below, and travels upward through a comparatively thick layer of incandescent fuel. The oxygen on coming into contact with the fuel is first converted into carbon dioxide, which on rising through the incandescent layer combines with more carbon, forming carbon monoxide. The heat generated volatilizes a certain amount of the bituminous coal in the upper layers from which the tarry matters escape unconsumed.

CRACKING OF OIL VAPORS

In manufacturing oil-gas, crude petroleum or a heavy distillate known as "gas-oil" is sprayed under more or less pressure into a closed retort heated to redness. This causes the oil to decompose into a permanent gas and from 5 to 10 per cent by weight of oil-gas tar. The reaction, known as "cracking," results in the breaking down of the hydrocarbons present in the petroleum or gas oil into simpler substances.

Water-gas is produced by the combustion of anthracite coal or coke in an atmosphere of steam according to the following reaction:

$$C + H_2O = CO + H_2$$

The gas consists theoretically of equal volumes of carbon monoxide and hydrogen. It burns with a non-luminous flame, and when intended for illuminating purposes must be enriched or "carbureted." The highly heated water-gas as it is generated, is accordingly mixed with a spray of crude petroleum or gas oil, then

passed into a carburetor in which the oil becomes vaporized, and finally through a superheater maintained at a temperature sufficiently high to crack the oil vapors into permanent gases. From 2 to 10 per cent of tar is produced, based on the weight of the petroleum or gas-oil used. This tar is known as water-gas tar and is similar in its physical and chemical properties to oil-gas tar.

CHAPTER XIV

WOOD TAR, WOOD-TAR PITCH AND ROSIN PITCH

WOOD TAR AND WOOD-TAR PITCH

This chapter will deal with the treatment of wood, either by destructive distillation, or by a combination of steam and destructive distillation.[1] The treatment of resinous woods by the steam distillation process alone, for the recovery of turpentine and other oils, does not fall within the scope of the present treatise.

Varieties of Wood Used. From the standpoint of destructive distillation, woods may be divided into two classes, viz.:

Hard Woods, including the maple, birch, beech, oak, poplar, elm, willow, aspen, alder, ash, hickory, chestnut and eucalyptus.

Resinous or Soft Woods, including the pine, fir, cedar, cypress, spruce, hemlock, larch or tamarack.

The trees from which hard woods are obtained are known as "broad-leaved" or "deciduous trees," and those producing resinous or soft woods are termed "coniferous trees" or "evergreens." Soft woods are distinguished from hard woods principally in that the former contain larger quantities of turpentine and resin. The distillation of hard wood aims at the recovery of wood alcohol, acetates, tar and charcoal, whereas the distillation of resinous wood (soft wood) is directed to the recovery of turpentine, wood-tar oils, tar and charcoal.

In the wood-distilling industry the basis of measurement is a cord, which is taken to equal 128 cu. ft. of the closely stacked wood containing 15 per cent of moisture. The weight of a cord varies with different kinds of wood, from about 1700 lb. in the case of white pine and poplar, to about 4000 lb. in the case of oak.

For purposes of destructive distillation, the wood should be as dry as possible, since during the process all the moisture must be evaporated before the wood decomposes. The smaller the percentage of moisture contained in the wood, the more rapid will be the distillation process and the smaller the quantity of fuel required.

It is advisable therefore, to cut and stack the green wood containing 20 to 50 per cent of moisture from six months to two years, during which the moisture content will fall to between 12 and 25 per cent.

Yields of Distillation. The following figures will give a general idea of the average yields upon distilling a cord of the respective classes of wood:

	Hard Woods	Soft (Resinous) Woods
Turpentine	0	5– 25 Gal.*
Wood-tar oils	0	30– 75 Gal.
Crude alcohol (containing acetone)	8– 12 Gal.	2– 4 Gal.
Tar	8– 20 Gal.	30– 60 Gal.
Charcoal	40– 52 Bu.	25– 40 Bu.
Acetate of lime	150–350 Lb.	50–100 Lb.

* Sawdust yields 5 to 10 gal. of turpentine and light wood 10 to 25 gal. per cord.

The tar may be classified according to the type of carbonizing apparatus employed, as follows: mound tar, pit tar, oven tar, retort tar and generator tar.

Hard-Wood Distillation. The following figures show the yields of tar and charcoal from the various hard woods in percentage, based on the dry weight of the material:[2]

	Tar, Per Cent	Charcoal, Per Cent
Hickory	13.0	37.7
Maple	12.8	40.6
Birch	12.0	40.6
Beech	9.4	41.9
Oak	7.8	45.7
Chestnut	4.6	47.6

In the United States, the principal centers for hard-wood distillation are in the States of Pennsylvania, New York and Michigan. Soft-wood distillation is carried on largely in the States of Florida, Georgia, North and South Carolina and Alabama.

The crude products of the distillation of hard wood may be grouped into four classes, viz.:

(1) Non-condensable gases..... 20–30 per cent
(2) Aqueous distillate (crude pyroligneous acid)..... 30–50 per cent
(3) Wood-tar oils, and wood tar..... 5–20 per cent
(4) Charcoal..... 20–45 per cent

Method of Distilling. When hard wood is heated in a retort, water passes off below 150° C., after which decomposition sets in. With soft (resinous) wood, turpentine and water commence to distil between 90 and 100° C. and continue to 150° C., whereupon products of destructive distillation pass over. The distillation process is practically complete at 430° C. In the case of hard wood, the first group of products which pass over (between 150 to 280° C.) include acetic acid, methyl alcohol and wood creosote; the second group (280 to 350° C.) consist of non-condensable gases (about 53 per cent of carbon dioxide, 38 per cent of carbon monoxide, 6 per cent of methane, 3 per cent of nitrogen, etc.); the third group (350 to 400° C.) are composed of solid hydrocarbons and their derivatives. The yields of methyl alcohol and acetic acid increase with a rise of temperature up to 300° C. beyond which they decrease; moreover their yield is greater when the wood is heated slowly, than when the distillation is forced.

The following fractions are obtained on distilling beech-wood tar (specific gravity at 15° C. 1.05–1.10):

Aqueous distillate (*)	15–20 per cent
Light tar oils (to 120° C.)	5–10 per cent (sp. gr. 0.90–0.98)
Heavy tar oils (120–270° C.)	10–20 per cent (sp. gr. 1.04–1.05)
Soft wood-tar pitch	40–60 per cent

* Containing 10 per cent acetic acid and 3–5 per cent crude wood alcohol.

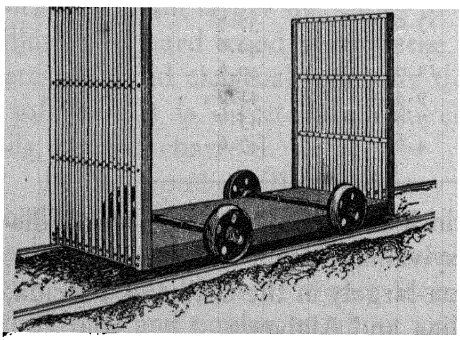


FIG. 91.—Iron Cars Used in the Distillation of Hardwood.

In distilling hard woods, large rectangular iron retorts are used, measuring 6 ft. in width, 7 ft. high and either 27 or 50 ft. long, depending upon whether they are intended to hold 2 or 4 carloads. The retorts are set in brickwork, and provided with large air-tight iron doors at the ends. The wood is loaded on small iron cars holding between 1 and 3 cords each (Fig. 91) which are run on tracks directly into the retorts.

The arrangement of a modern wood-distilling plant is shown in Fig. 92; where *A* represents a car; *B*, the retort; *C*, first cooler; *D*,

second cooler; *E*, the acetate drying floor; *a*, condensers; *b*. liquor trough; *c*, gas main to boilers; *i*, fuel conveyor; *m*, fireplace; *n*, ash pit; *o*, hinged spout to deliver fuel from *i* to *m*. After the retort is charged, the doors are closed and heat applied slowly, either by

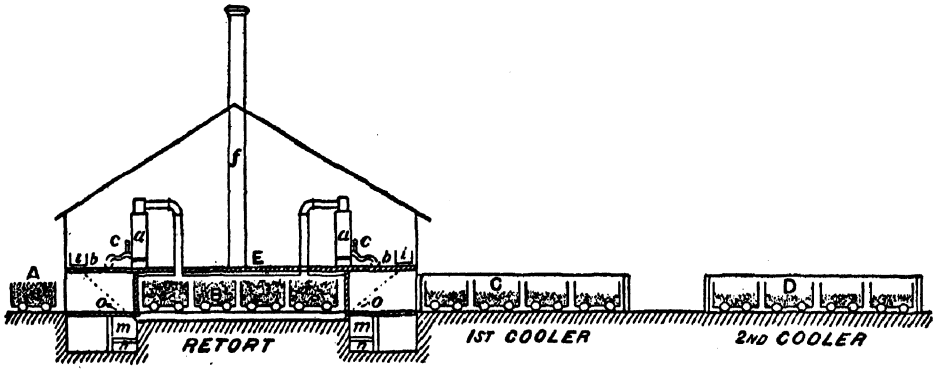


FIG. 92.—Modern Wood Distilling Plant.

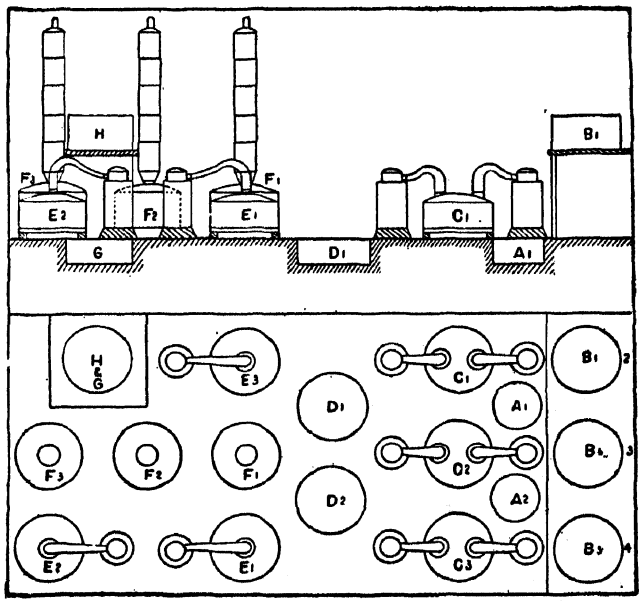


FIG. 93.—Plant for Refining Wood Tar.

burning the non-condensable gases resulting from the distillation process, or by atomizing the tar underneath the retort with a jet of steam. Unless the gases are stored in a gas-holder, the process is started by burning a small amount of wood on an auxiliary grate beneath the retort.

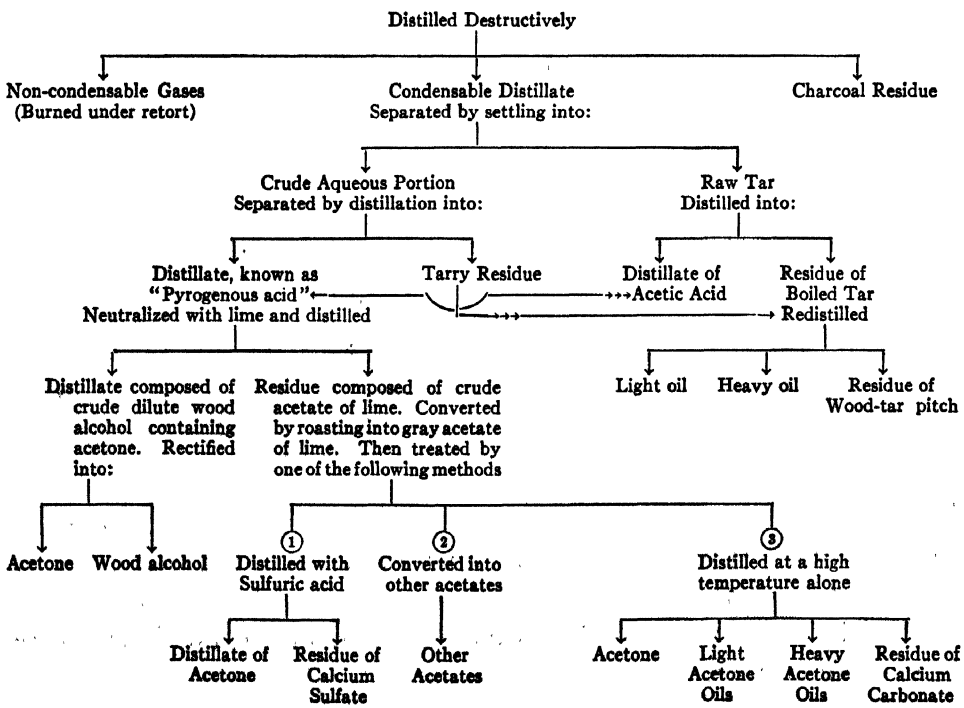
The vapor from the retort is passed through condensers, where the pyroligneous acid, alcohol and other condensable constituents are recovered. These are conveyed to large settling tanks, and allowed to rest quietly until the tar settles out.

The distillation process continues from twenty to thirty hours, whereupon the fires are extinguished and the retort allowed to cool. The small iron cars now carrying charcoal are quickly run from the retort into large iron coolers, similar in size and shape to the retort itself, and the doors are closed to prevent access of air.

Refining Processes. The general arrangement of a refining plant is shown in Fig. 93, where *A* 1–2 represents the raw liquor vats, *B* 1–5 represent the raw liquor settling tanks, *C* 1 the tar still, *C* 2–3 the raw liquor still, *D* 1–2 the neutralizing vats, *E* 1–3 the lime-lee stills, *F* 1–3 the alcohol stills, *G* the weak alcohol storage tank and *H* the strong alcohol storage tank. Table XVII shows a diagrammatic outline of the products obtained upon distilling hard wood, and refining its distillates.

TABLE XVII

HARD WOOD



After the pyroligneous acid and tar have been separated by settling, the crude products are distilled independently to recover any pyroligneous acid from the crude tar, and conversely, any tar retained by the crude pyroligneous acid (dissolved in the alcohol and acetone present). The method consists in heating the tar with steam in closed coils until the water is driven off, and then blowing live steam through the charge.[3]

A continuous process, known as the "Badger-Stafford Process," [4] has been devised for treating scrap wood obtained in other manufacturing operations. The hogged scrap wood (averaging 70 per cent maple, 25 per cent birch and 5 per cent ash, elm and oak) is first passed through six rotary driers heated by flue gases entering at 600° F. The scrap wood is discharged from the driers at 302° F. and then fed into retorts 40 feet high and 10 feet in diameter. Air-tight valves at the top and bottom allow the introduction of the wood and the removal of the charcoal, respectively, without escape of gas, which passes off at the top of each retort to four condensers mounted around it. The average temperature in the central zone is 950° F., and at the bottom 490° F.

The products from the retorts are charcoal, which is passed to coolers and conditioners; pyroligneous acid or "green liquor," which is pumped from the condensers to the primary separators; and non-condensable gas, which is used for heating the retorts and burned under the boilers.

Each ton of wood produces 111 gal. of pyroligneous acid containing approximately 4.5 per cent methanol and 12.5 per cent acetic acid, also allyl alcohol, acetone, methyl acetate, suspended and dissolved tar, various oils and water. The pyroligneous acid is first delivered to settling tanks where the suspended tar is largely removed, and then pumped to the primary tar stills. The acid distillate from these stills is returned to the settling tanks, while the distilled heavy tar is sent to the pitch still, where creosote oil and pitch are recovered by distillation under a vacuum of 27 inches with the use of steam at 125 lb. pressure.

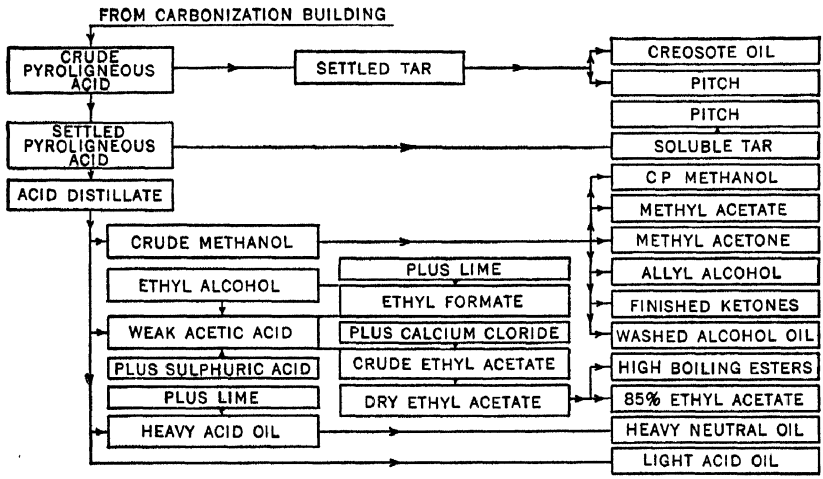
The pyroligneous acid free of settled tar is distilled in six large batch primary stills operating on a 30 hour cycle. The soluble tar remaining as a residue in these stills is pumped under the boilers where it is burned. The acid distillate passes to a separator where

the acid oils are removed, then through a sand filter, and finally to the continuous stills, which yield crude 95 per cent methanol at the top of the column and 15 per cent acetic acid at the bottom. The crude methanol, which contains allyl alcohol, acetone, methyl acetate and alcohol oils, is first fractioned in two discontinuous stills, which yield refined allyl alcohol and alcohol oils as final products, while a 50 per cent methyl acetone, an intermediate methyl acetate, and a 99 per cent methanol are sent to the four-column continuous stills, which in turn yield 75 per cent methyl acetone, 75 per cent methyl acetate and C.P. methanol.

The 15 per cent acetic acid obtained from the first continuous de-alcoholizing distillation is used directly in the production of ethyl acetate from ethyl alcohol in the presence of sulfuric acid.

A flow-sheet of the process is illustrated in Table XVIII.

TABLE XVIII



The following table gives the yields of the various intermediate and final products per ton of dried wood treated:

Intermediate Products		Final Products	
		Charcoal, lbs.	600
		Non-condensable gas, cubic feet (290 B.t.u. per cubic foot)	5000
Total 100% spirits, gallons	5.0	C. P. methanol, gallons	3.118
		Methyl acetone, gallons	0.653
		Allyl alcohol, gallons	0.048
		Ketones, gallons	0.226
		Methyl acetate, gallons	0.945
		Soluble tar, gallons	22.0

Settled tar, gallons	11.0	Pitch, lbs.	66.0
		Creosote oil, gallons	3.25
Acetic acid (as 100%), lbs.	101.0	Ethyl acetate, gallons	14.65
		Ethyl formate, gallons	1.27

Various other processes have been devised for distilling wood waste in the production of wood tar involving the treatment of sawdust, shavings, splittings, wood bark, roots, refuse of one kind or another.[5]

The dehydrated tar, known as "boiled tar" or "retort tar," amounting to between 3 and 10 per cent of the weight of the wood, may be utilized in one of the following ways:

(1) It may be sold as such, and used for preserving wood.
(2) It may be burned under the retorts as fuel.
(3) It may be subjected to fractional distillation to recover the light oils boiling below 150° C., heavy oils boiling between 150° and 240° C., and the residual pitch constituting between 50 and 65 per cent by weight of the tar. The light oils are used as solvents in manufacturing varnish, and the heavy oils after further refining are marketed as commercial wood creosote which finds a sphere of usefulness as a disinfectant, wood preservative, and flotation oil.

Soft (Resinous) Wood Distillation. *Method of Distilling.* In treating soft wood (resinous wood) a different method is followed. Iron or steel retorts varying in capacity from one to four cords are used, constructed either vertically or horizontally, as shown in Fig. 94. Low-pressure superheated steam, or saturated steam under high pressure, is introduced into the retort to remove the turpentine, and then the volatile oils (known as "heavy oils") leaving a residue of coke behind. Three classes of resinous wood are used for the purpose:

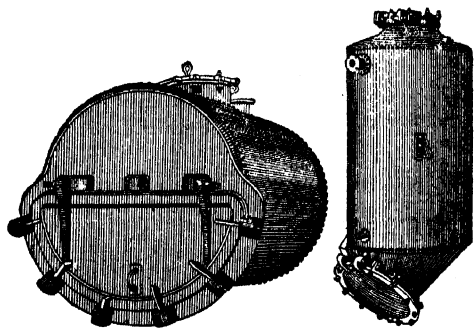


FIG. 94.—Retorts for Distilling Soft Wood.

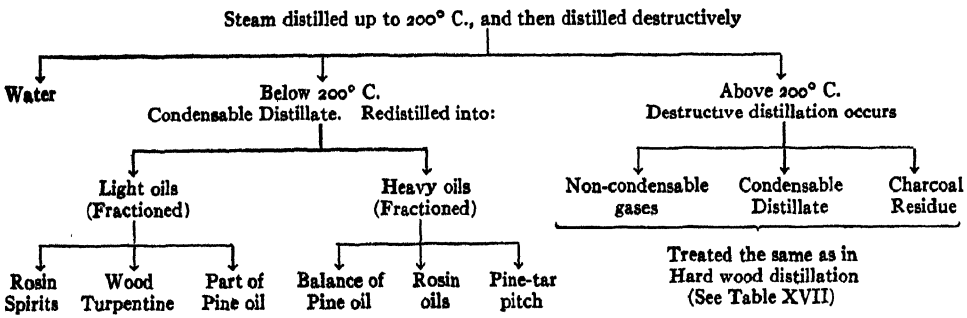
(1) "Light wood" containing comparatively large quantities of turpentine.

(2) "Stumps," which also contain more or less turpentine
(3) Saw-mill waste, which is rather poor in turpentine.

The wood is first "hogged," or in other words, cut into chips before it is introduced into the retort. The temperature is raised gradually to 200° C. as the steam passes through the retort. Water and crude turpentine distil over first and are separated by settling. As the temperature rises above 200 to 220° C. the wood commences to decompose into tarry substances, and at about 250° C. the resins present break up into "rosin spirits" and "rosin oils." Both the crude turpentine and the heavy oils are redistilled separately, the former producing purified wood turpentine and the latter pine oil, rosin oil and pitch. Rosin spirits boils between 80 and 200° C., wood turpentine between 150 and 180° C., pine oil between 190 and 240° C., and rosin oil between 225 and 400° C. This process is illustrated diagrammatically in Table XIX.

TABLE XIX

LIGHT WOOD (PINE OR RESINOUS WOODS)



In some cases the "light wood" is subjected to a process of destructive distillation without using steam. The temperature is raised slowly and the distillate under 200° C. caught separately to avoid contamination with tarry matters. After the temperature rises above 200° C., the process follows the same course as for hard-wood distillation.

Refining Processes. The distillate under 200° C. is fractioned into light and heavy oils respectively. The light oil is in turn redistilled to recover the rosin spirits, wood turpentine and a part of the

pine oil. The heavy oil is similarly redistilled to separate the pine oil, rosin oil and a part of the pitch. The crude tar obtained above 200° C. is distilled to recover any acetic acid, and the residue either marketed as "pine tar" or distilled to separate the light and heavy oils from the pine-tar pitch obtained as residue. A pitch-like product may be obtained upon blowing rosin oil with air and superheated steam.[6]

Another treatment of soft wood consists in subjecting it to an extraction process with solvents, which are recovered and used over again. In this process, the wood is first ground into small fragments, which may either be treated as such, or else subjected to a treatment with live steam to remove the volatile oils, including the turpentine and pine oil. The rosin is extracted with hot petroleum naphtha or benzol. The extract is thereupon distilled to remove the solvent (also the turpentine and pine oil, where the wood was extracted directly, without steaming). The rosin is extracted from the residue by means of a petroleum solvent (sometimes after first purifying by dissolving in furfural and subsequent evaporation), leaving a form of wood pitch which tests as follows:

(Test 15*b*)	Fusing-point (R. and B. method)	195–240° F.
(Test 23)	Soluble in 88° petroleum naphtha	10–20 per cent.
(Test 37*a*)	Acid value	11–115
(Test 37*d*)	Saponification value	140–170
(Test 37*e*)	Saponifiable constituents	92–95 per cent.

The wood, after treatment in this manner, may either be distilled destructively, or used as a fuel, or used as a filler in other manufacturing operations.

Wood Tars. The bituminous products derived from the destructive distillation of wood are designated commercially as hard-wood tar and pine tar; hard-wood-tar pitch and pine-tar pitch.

Wood tar has also been termed "Stockholm tar" and "Archangel tar."

Hard-wood Tar and Pine Tar. The physical and chemical characteristics of the tars and corresponding pitches vary, depending upon the kind of wood used, as well as the exact method of treatment. The following figures will give a general idea of the

characteristics of the dehydrated hard-wood tar and pine tar ordinarily encountered in the American market:

		Hardwood Tar	Pine Tar (from Resinous Woods)
(Test 1)	Color in mass	Black	Brownish
(Test 7)	Specific gravity at 77° F.	1.10–1.20	1.05–1.10
(Test 8)	Viscosity	Fairly liquid	Viscous
(Test 9)	Consistency at 77° F.	Liquid	Liquid
(Test 15)	Fusing-point	Below 20° F.	Below 50° F.
(Test 16)	Volatile matter at 500° F., 5 hrs.	35–60 per cent	40–75 per cent
(Test 17*a*)	Flash-point	50–75° F.	60–90° F.
(Test 19)	Fixed carbon	5– 20 per cent	5– 15 per cent
(Test 21)	Solubility in carbon disulfide	95–100 per cent	98–100 per cent
	Non-mineral matter insoluble	0– 5 per cent	0– 2 per cent
	Mineral matter	0– 1 per cent	0– 1 per cent
(Test 22)	Carbenes	0– 2 per cent	0– 2 per cent
(Test 23)	Solubility in 88° petroleum naphtha	50– 90 per cent	65– 95 per cent
(Test 28)	Sulfur	0.0 per cent	0.0 per cent
(Test 30)	Oxygen	2–10 per cent	5–10 per cent
(Test 32)	Naphthalene	None	None
(Test 33)	Solid paraffins	None	None
(Test 34*a*)	Sulfonation residue	Trace to 5%	Trace to 5%
(Test 37*e*)	Saponifiable constituents	25–85%	20–60%
(Test 37*g*)	Resin acids	Up to 15%	Up to 30%
(Test 39)	Diazo reaction	Yes	Yes
(Test 40)	Anthraquinone reaction	No	No
(Test 41)	Liebermann-Storch reaction	Yes	Yes

According to David Holde, on shaking wood tar with water, the aqueous extract will react acid (due to the acetic acid present), and upon adding a few drops of ferric chloride, will at first form a green and then a bluish- to brownish-green coloration. Birch-bark tar gives a green color with ferric chloride, which on addition of ammonia changes to an intense blue; whereas birch tar gives a brown color. Both hard-wood tar and pine tar are almost completely soluble in absolute alcohol, glacial acetic acid and acetic anhydride. On subjecting wood tar to distillation, the first portion passing over shows a separation of water which will react acid. On continuing the distillation, oily matters are obtained which dissolve readily in alcohol, and on treatment with concentrated sulfuric acid become converted into water-soluble substances. Pine tar has a high acid value, since it often contains as much as 30 per cent by weight of resin acids, and is characterized by the absence of sulfur, paraffins, naphthalene and anthracene; and by the presence of phenol (Test 39) and resin acids (Test 41) which causes it to have a high acid value (Test 37*a*) and give an acid reaction to litmus.

The following constituents have been identified in German tars:[7]

	Beechwood Tar Per Cent	Pine Tar Per Cent
Unsaponifiable matter	18.0	53.5
Saponifiable matter:		
Hydroxy acid anhydrides	9.5	0.0
Hydroxy acids	52.3	14.0
Resin acids	7.7	17.0
Reported as fatty acids*	3.2	6.0
Phenols	9.3	9.5
Total	100.0	100.0

* Separated by Twitchell's method, but not true fatty acids.

Wood-tar Pitches. Hard-wood-tar pitch and pine-tar pitch vary in their physical properties, depending upon the following circumstances:

(1) The variety of wood used.
(2) The method by which the wood is distilled, including the temperature, its duration, the kind of retort, etc.
(3) The extent to which the tar is distilled in producing the pitch. The further it is distilled, the harder will be the pitch and the higher its fusing-point.

Hardwood-tar Pitch and Pine-tar Pitch. These comply with the following characteristics:

		Hardwood-tar Pitch	Pine-tar Pitch (from Resinous Wood)
(Test 1)	Color in mass	Black	Brownish black
(Test 2)	Homogeneity	Uniform	Uniform
(Test 4)	Fracture	Conchoidal	Conchoidal
(Test 5)	Lustre	Bright to dull	Bright to dull
(Test 6)	Streak	Brown to black	Brown
(Test 7)	Specific gravity at 77° F.	1.20–1.30	1.10–1.15
(Test 9*b*)	Penetration at 77° F.	0–60	0–60
(Test 9*c*)	Consistency at 77° F.	10–100	10–100
(Test 9*d*)	Susceptibility index	> 100	> 100
(Test 10)	Ductility	Variable	Variable
(Test 15*a*)	Fusing-point (K. and S. method)	100–200° F.	100–200° F.
(Test 15*b*)	Fusing-point (R. and B. method)	115–225° F.	115–225° F.
(Test 16)	Volatile matter	Variable	Variable
(Test 19)	Fixed carbon	15–35 per cent	10–25 per cent
(Test 21)	Soluble in carbon disulfide	30–95 per cent	40–95 per cent
	Non-mineral matter insoluble	5–70 per cent	2–60 per cent
	Mineral matter	0–1 per cent	0–1 per cent

		Hardwood-tar Pitch	Pine-tar Pitch (from Resinous Wood)
(Test 22)	Carbenes	2–10 per cent	0–5 per cent
(Test 23)	Solubility in 88° petroleum naphtha	15–50 per cent	25–80 per cent
(Test 28)	Sulfur	0 per cent	0 per cent
(Test 30)	Oxygen in non-mineral matter	1–5 per cent	2–8 per cent
(Test 32)	Naphthalene	None	None
(Test 33)	Solid paraffins	None	None
(Test 34*a*)	Sulfonation residue	0–5 per cent	0–3 per cent
(Test 37*e*)	Saponifiable constituents	60–95 per cent	45–75 per cent
(Test 37*g*)	Resin acids	Up to 20 per cent	Up to 40 per cent
(Test 39)	Diazo reaction	Yes	Yes
(Test 40)	Anthraquinone reaction	No	No
(Test 41)	Liebermann-Storch reaction	Yes	Yes

The following constituents have been identified in German pitches:[8]

	Hard Beechwood-tar Pitch, Per Cent	Medium Hard Pine-tar Pitch, Per Cent
Unsaponifiable matter	6.0– 6.3	19.7
Saponifiable matter:		
Hydroxy acids and anhydrides	77.0– 65.3	31.8
Resin acids	0.0– 0.0	35.2
Neutral tar resins	14.0– 25.4	1.5
Reported as fatty acids *	1.5– 1.5	2.8
Phenols	1.5– 1.5	8.0
Mineral matter	0.0– 0.0	1.0
Total	100.0 100.0	100.0

* Separated by Twitchell's method, but not true fatty acids.

Soft and medium hard wood-tar pitches are fairly soluble in ethyl alcohol, and as the hardness increases the solubility decreases. Hard pitches are only sparingly soluble.

According to Benson and Davis,[9] wood-tar pitches are more soluble in acetone than in carbon disulfide. Thus, hardwood-tar pitches were found to be 15.6–31.9 per cent more soluble in acetone than in carbon disulfide, and pine-tar pitches (obtained from the Douglas fir) 8.0 to 57.8 per cent more soluble in the former solvent.

Wood-tar pitches are characterized by their extreme susceptibility to changes in temperature, by the fact that they appear hard and at the same time show a surprisingly low fusing-point,[10] and are characterized by the absence of sulfur, paraffins, naphthalene and anthracene. Wood-tar pitches are notoriously non-weatherproof. They are extremely susceptible to oxidation on exposure to the weather and are soon converted into a lifeless and pulverulent mass.

However, their physical properties and weather-resistance may be improved by fluxing with cashew-nut-shell oil,[11] or by blending with fatty-acid pitch, which latter may be accomplished by heating together pitches of the same fusing-points, respectively.[12]

Wood tar may be hardened and made more suitable for use in black paints by treating with concentrated sulfuric acid removing the water-soluble substances,[13] and the product may be dissolved in acetone or methyl alcohol for use as lacquers.[14] Artificial resins may also be produced by treating wood tar with formaldehyde in the presence of a catalyst,[15] or by heating wood tar with 10 per cent zinc oxide at 110° C.,[16] or by treatment with chlorine in the presence of $AlCl_3$ as catalyst,[17] in which latter case the product is soluble in methyl alcohol. Wood tars may be hardened by heating with sulfur,[18] or by blowing with air in the presence of a small amount of sulfur,[19] or by blowing with air, ozonized air or oxygen at 120–150° C.[20]

Wood tars have been utilized for the production of tar soaps, lubricants, flotation agents, pharmaceutical products, etc.[21]

ROSIN PITCH

Raw Materials Used. The sap of the long-leaf pine, known chemically as an oleo-resin, is composed of a mixture of spirits of turpentine and rosin. It is gathered by incising the bark one-half to one inch, whereupon the oleo-resin slowly exudes and is collected in small cups.

The oleo-resin is then distilled to separate the spirits of turpentine from the rosin. The apparatus ordinarily used in the United States for this purpose is shown in Fig. 95, consisting of a simple type of copper still with a "worm" condenser. The capacity of the still varies from 10 to 40 barrels, and usually between 15 and 20. After the still is charged, the fire is started, and a mixture of spirits of turpentine and water (since the oleo-resin contains between 5 and 10 per cent of water) appears in the condenser. When all the water has boiled over, additional quantities are added in a small stream during the distillation, since the introduction of water causes the turpentine to boil at a lower temperature and prevents overheating, improving both the color and yield of the turpentine and rosin. Towards the end of the distillation the stream of water is

shut off, and the rosin heated until all the moisture is expelled, usually between 300 and 400° F. Before cooling, any foreign matter is skimmed off the surface of the rosin, after which it is strained through a fine mesh screen and barreled.[22]

Method of Distilling. Rosin, deprived of its turpentine, when heated in a closed retort undergoes destructive distillation, yielding a gas, an aqueous liquor and an oily distillate which may be separated into several fractions. If the process is carried to completion, coke will be left as residue. If the distillation is terminated before the formation of coke, a pitchy residue will remain, known commercially as "rosin pitch."

The rosin may be distilled either with or without superheated steam. If the latter is employed, the quality of the distillate is im-

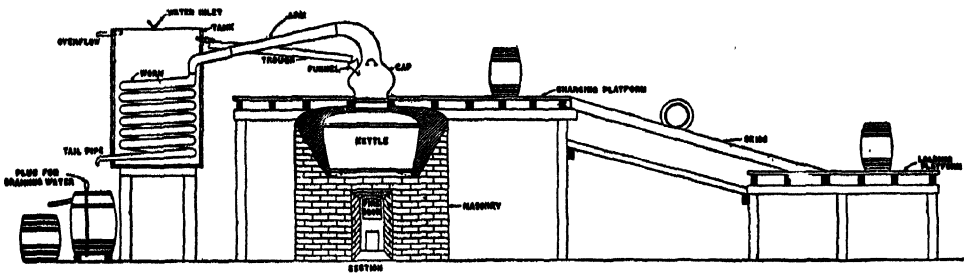


FIG. 95.—Retort for Distilling Rosin.

proved, and a much better temperature control obtained. Distillation under vacuum is also used in many cases. The rosin may accordingly be distilled by any of the following processes:

(1) At atmospheric pressure without steam.
(2) With superheated steam.
(3) Under vacuum.

When the temperature of the rosin reaches 150° C. a liquid distillate appears which separates into two layers, the lower containing acetic acid, also other organic acids dissolved in water, and the upper composed of oily substances known as "rosin spirits" or "pinoline." When the temperature reaches 200° C. the receiver is changed, and the distillate which ensues is either collected together or separated into fractions. The temperature of the residue in the retort is permitted to reach 350 to 360° C. but never to exceed the latter. The distillate between 200 and 360° C. known as "rosin oil," may be separated into various fractions termed "yellow rosin

oil," "blue rosin oil," "green rosin oil," etc., depending upon their respective colors.

Products Obtained. In distilling rosin destructively at atmospheric pressure, the following products are separated:

Non-condensable gases	9.0 per cent
Acid liquor	3.5 per cent
Rosin spirits or pinoline	3.5 per cent
Rosin oil	67.0 per cent
Rosin pitch	16.0 per cent
Loss (rosin adhering to walls of still, etc.)	1.0 per cent

According to Victor Schweizer,[23] when rosin is distilled with superheated steam, the following yields are obtained:

Acid liquor	5.5– 5.8 per cent
Rosin spirits	11.25–12.0 per cent
Blue rosin oil	49.0 –50.5 per cent
Brown rosin oil	10.25–10.65 per cent
Rosin pitch	18.0 –19.0 per cent

The rosin pitch is run from the still while it is hot, and allowed to cool in a suitable receiver.

Properties of Rosin Pitch. It is fairly uniform in composition and conforms with the following characteristics:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	Uniform
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Dull
(Test 6)	Streak	Light yellow to brown
(Test 7)	Specific gravity at 77° F.	1.08–1.15
(Test 9*b*)	Penetration at 77° F.	0.5
(Test 9*c*)	Consistency at 77° F.	50–100
(Test 9*d*)	Susceptibility index	Greater than 100
(Test 10*c*)	Ductility at 77° F.	0
(Test 15*a*)	Fusing-point (K. and S. method)	120–200° F.
(Test 15*b*)	Fusing-point (R. and B. method)	135–225° F.
(Test 16)	Volatile matter, 500° F., 5 hrs.	10–18 per cent
(Test 17)	Flash-point	Above 250° F.
(Test 19)	Fixed carbon	10– 20 per cent
(Test 21)	Soluble in carbon disulfide	98–100 per cent
	Non-mineral matter insoluble	0– 2 per cent
	Mineral matter	0– 1 per cent
(Test 22)	Carbenes	0– 5 per cent
(Test 23)	Solubility in 88° petroleum naphtha	90–100 per cent
(Test 28)	Sulfur	0.0 per cent
(Test 30)	Oxygen in non-mineral matter	5– 10 per cent
(Test 33)	Solid paraffins	0.0 per cent
(Test 34*a*)	Sulfonation residue	0– 5 per cent
(Test 37*e*)	Saponifiable constituents	25–95 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	Yes

Rosin pitch is very much like rosin in its physical properties. It is extremely susceptible to temperature changes, and as ordinarily produced, is hard and friable at 77° F. It is characterized by the presence of considerable quantities of unaltered resin acids (10 to 45 per cent), and is free from fatty acids, glycerol, sulfur and paraffin. It withstands weathering very poorly, and has therefore but a limited use, including the lining of barrels and casks ("brewers' pitch"). Upon being heated, it passes rapidly from the solid to the liquid state, forming a melt of low viscosity. A product similar to rosin pitch may be produced by heating rosin with sulfur at 250° C.[24]

"Burgundy pitch" is the name applied to the oleo-resin which exudes from the Norway spruce (*Abies excelsa*), found in the Vosges Mountains and in the Alps; also from a species of pine obtained in the United States (*Pinus australis*). The crude oleo-resin is melted by boiling with water, and strained to remove any particles of bark or other impurities. It then constitutes the so-called "Burgundy pitch" (*Pix abietina*), sometimes marketed under the name "Vosges pitch." These terms are misnomers, since the material is not a true "pitch," but in reality an oleo-resin. It contains more or less spirits of turpentine, which escaped expulsion during the boiling process, also a quantity of emulsified water imparting to it an *opaque*, yellowish-brown color. In consistency it is a more or less brittle solid, largely susceptible to temperature changes. In summer it softens and gradually flows, and in winter it appears very hard and brittle. It melts easily, decrepitating because of the water present, and has a strong odor because of the associated spirits of turpentine. On aging it loses its opacity, due to evaporation of the emulsified water, and turns first to a translucent, and then to a transparent brown color, similar to that of rosin. Its composition is substantially the same as rosin, containing in addition, spirits of turpentine and emulsified water.

CHAPTER XV

PEAT AND LIGNITE TARS AND PITCHES

PEAT TAR AND PEAT-TAR PITCH

Formation of Peat. Peat[1] is derived from the decomposition of vegetable matter in swampy places, such as marshes and bogs. On the surface we find the growing aquatic plants; somewhat deeper we find their decayed remains; and still deeper a dark colored pasty substance containing a substantial percentage of moisture and constituting the crude peat. The plants which result in the formation of these deposits are mainly aquatic, including marine grasses, reeds, rushes, hedges and various mosses. The transformation is caused partly by oxidation in the presence of moisture, and also to some extent by the action of certain forms of bacteria, molds and fungi. As the mass of peat builds up in thickness, the lower layers are first compacted by the resulting pressure, and then gradually carbonized. The essential condition to peat formation is that the vegetable remains shall be deposited at a rate exceeding that of their decomposition. This does not prove to be the case in very warm climates, where the remains are entirely decomposed. The organic matter should only be *partly* decomposed, and since the products of partial decomposition act as a preservative to inhibit further decay, we can readily understand why the building up of peat beds is cumulative. It progresses most rapidly at a mean atmospheric temperature of 45° F., which accounts for the fact that no peat bogs occur between the latitudes of 45° N., and 45° S. It is estimated that there exist in the United States 20 million acres of peat bogs, 30 million acres in Canada, 50 million on the continent of Europe, also approximately 3 million in Ireland.

Varieties of Peat. The following constitute the most important varieties of peat, based on the locality in which they are found:

(1) "Hill peat," found at mountain tops and derived from plants consisting of sphagnum and andromeda mosses, likewise heath.

(2) "Bottom peat," found near rivers, lakes, etc., in the lowlands, including: (*a*) dark peat, approaching lignite in composition, occurring at the lower parts of the deposit; (*b*) middle peat, which is lighter in color and in weight than the preceding; (*c*) the top stratum, which has a fibrous structure.

Peat varies in color from light yellowish, through various tints of brown, to brownish black or black, all of which appear deeper when the peat is moist. The lighter shades generally darken to brownish black or black upon exposure to air; due largely to oxidation. In texture, peat varies from light porous matter having a fibrous or woody structure, to substances which are amorphous and clay-like when wet, but appearing quite hard and dense upon drying. When recently formed, the peat beds are but loosely compacted, but as they accumulate, the under layers become compressed, so what once was a foot thick may be concentrated to several inches. In other cases the beds become covered with sedimentary rocks, which augment the pressure, and gradually transform the peat into lignite.

The chemical composition of peat is but little understood. It is regarded as a mixture of water, inorganic matter (calcium and iron compounds), vegetable fibers and humus acids (such as humic, ulmic, crenic, apocrenic, etc.). According to H. Bornträger [2] the black varieties contain between 25 and 60 per cent of humic acids, 30 to 60 per cent of fiber, and 3 to 5 per cent of ash. Nitrogenous compounds are also present, varying from 1 to 3 per cent of the dry weight, resulting partly from the associated animal matter, and also due partly to the humic acids combining with atmospheric nitrogen, forming what are known as azo-humic acids. Sulfur is also present in amounts between 0.1 and 5.3 per cent based on the dry weight.

Resinous substances are found in certain varieties of peat, to which various names have been assigned, also bodies of a waxy nature derived from the associated gelatinous algae, known as "sapropel." In time, sapropel is converted into a coal-like substance, known as sapropelite, or sapropelite coal, which has been regarded as the progenitor of cannel coal and boghead coal.

Methods of Collecting Peat. Peat is generally collected by cutting trenches through the bog with a spade, and removing it in sods about 3 to 4 ft. long. The deposits are worked in steps or

tiers. Mechanical excavators and dredges have also been used for the purpose. The sods are allowed to drain, then air-dried and finally heated to a high temperature in either stationary or revolving ovens, to remove the water. Peat as freshly mined contains 75 and 90 per cent by weight of water, which must of necessity be removed before the product can be used as a fuel. Air-dried peat carries 10 to 15 per cent of moisture, and the artificially dried peat between a trace and 80 per cent of mineral ash, consisting principally of sand and clay with smaller quantities of iron oxide, calcium and magnesium salts. The maximum quantity of ash usually considered allowable when used as a fuel is 25 per cent of the dry weight. Peat with less than 5 per cent ash is considered good, between 5 and 10 per cent as fair, and over 10 per cent as poor, with peat containing less than 10 per cent of ash in the moisture-free state, the fixed carbon varies between 15 and 35 per cent, averaging about 30 per cent.

Dehydrating Processes. It is customary to briquette the partly dried peat, carrying 10 to 15 per cent of water, and then continuing the drying until practically all the moisture is removed, and the residual peat compacted into tough briquettes suitable for use as fuel. It is briquetted under a pressure of 18,000 to 30,000 lb. per square inch, which generates sufficient heat to liberate some of the tarry compounds of the peat, causing the sides of the briquettes to assume a highly polished glaze. The product is claimed to have a calorific value almost equal to that of coal. Various mechanical contrivances have been devised for removing the water and moisture from peat.

In Europe and Canada, attempts have been made to utilize air-dried peat for generating producer gas. Various types of apparatus have been devised for the purpose, which result in the recovery of 1 to 2 per cent of peat tar, based on the dry weight of the peat.[8] In generators (e.g., Mond gas generators) from 8 to 9 per cent of tar is recovered, and in low temperature distillation processes from 8 to 25 per cent is recovered.

Methods of Distilling. Various methods have been used for distilling peat, similar to those employed for treating coal. Dried peat may be destructively distilled in closed retorts, obtaining a gas suitable for use as a fuel, likewise tar, ammonia, and a good grade

of coke, but in the United States this process has only been carried on in a small experimental way. There are two classes of peat tar, namely retort peat tar and producer peat tar, both of which are low-temperature products generated at not exceeding 600° C. At the present time the cost of drying and briquetting peat brings its price higher than that of bituminous coal. For these reasons neither peat tar nor peat-tar pitch are produced in commercial quantities.

The following represent the percentages by weight of by-products obtained per ton of the air-dried peat:

Gases and loss	10–20 per cent
Aqueous liquor	30–40 per cent
Peat tar	2–10 per cent
Coke	30–40 per cent
	100 per cent

The composition of an average specimen of peat tar is as follows:

Insoluble in ether (i.e., oxy-acids and their esters, also iron and calcium derivatives)	3 per cent
Soluble in ether:	
Bases	5 per cent
Unsaponifiable substances:	
Solid portion	8 per cent
Liquid portion	55 per cent
Saponifiable substances:	
Oxy-acids—	
Insoluble in ether	3 per cent
Soluble in ether	8 per cent
Fatty acids	10 per cent
Phenols	3½ per cent
Water	4½ per cent
Total	100 per cent

The following yields are obtained from German peat:

Water-free peat tar	4.9 to 9.0 per cent
Light oils (sp. gr. 0.820–0.835)	7.3 to 34.6 per cent
Heavy oils (sp. gr. 0.830–0.885)	19.6 to 36.0 per cent
Paraffin	3.3 to 46.0 per cent
Peat-tar pitch	12.8 to 22.6 per cent
Creosote oil and loss	6.2 to 40.5 per cent

Refining Processes. The tar is separated from the aqueous liquor by heating the mixture with steam to the melting-point of the tar, which then rises to the surface. It is a black, viscid liquid with a disagreeable acrid odor, representing 2 to 20 per cent of

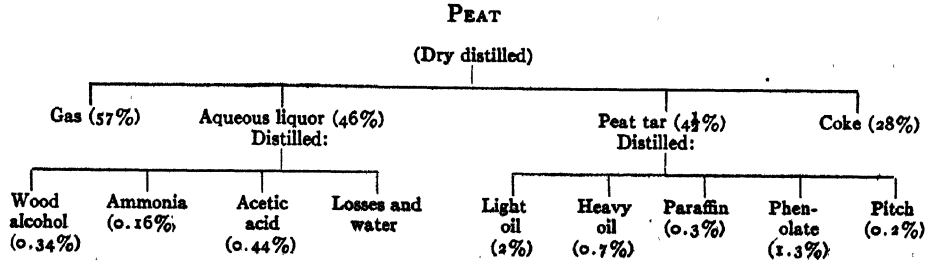
the dry weight of the peat used. The aqueous liquor contains ammonia salts, acetic and other organic acids, wood alcohol, and pyridine bases. The tar is slowly distilled, and after the water ceases to pass over, the receiver is changed and the distillation continued until 45 per cent of oily distillate has been collected. The receiver is again changed, and heavy oils containing paraffin wax, totalling about 30 per cent by weight of the tar, are caught separately, leaving 15 to 20 per cent of peat-tar pitch in the retort, which is finally drawn off. The oily distillate first obtained is redistilled into light naphtha (density under 0.83), and heavy naphtha (density 0.85). The heavy oil is cooled and pressed to separate lubricating oil from the paraffin wax. The products are treated first with concentrated sulfuric acid and then with caustic soda to remove tarry impurities and creosote oil respectively, the latter being recovered in the form of creosote or carbolic acid.

The dry peat tar yields the following:

	Crude, Per Cent	After Purification, Per Cent
Light naphtha	16	12
Heavy naphtha	30	25
Lubricating oil	15	13
Paraffin wax	12	2
Peat-tar pitch	16	16
Creosote	..	12
Loss	11	20
	100	100

A diagrammatical outline of the various products obtained on the dry-distillation of peat is given in Table XX.

TABLE XX



Properties of Peat Tar. Dehydrated peat tars in general, test as follows:

(Test 1)	Color in mass	Black
(Test 7)	Specific gravity at 77° F.	0.90–1.05
(Test 9)	Hardness or consistency	Liquid
(Test 15*a*)	Fusing-point (K. and S. method)	40–60° F.
(Test 16)	Volatile matter at 500° F., in 5 hrs.	50–85 per cent
(Test 17*a*)	Flash-point	60–95° F.
(Test 19)	Fixed carbon	5–15 per cent
(Test 21)	Soluble in carbon disulfide	98–100 per cent
	Non-mineral matter insoluble	0– 2 per cent
	Mineral matter	0– 1 per cent
(Test 22)	Carbenes	0– 2 per cent
(Test 23)	Solubility in 88° petroleum naphtha	95–100 per cent
(Test 28)	Sulfur	Less than 1 per cent
(Test 30)	Oxygen in non-mineral matter	5–15 per cent
(Test 33)	Solid paraffins	5–15 per cent
(Test 34*b*)	Sulfonation residue	5–15 per cent
(Test 37*e*)	Saponifiable constituents	5–15 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	No

Properties of Peat-tar Pitch. Peat-tar pitch is obtained by the evaporation or steam distillation of peat tar. It is not an article of commerce in the United States. Its hardness or consistency, as well as its fusing-point, depend upon the extent to which the distillation has been conducted. Ordinarily, peat-tar pitch tests much the same as lignite-tar pitch, the results being included in Table LXXXIV. It is highly susceptible to temperature changes, and withstands exposure to the weather very poorly.

LIGNITE TAR AND LIGNITE-TAR PITCH[4]

Varieties of Lignite. The U. S. Geological Survey estimates that 1,087,514,400,000 tons of lignite are available in the United States, but it is used only in a limited way, due to the abundance of other types of fuel. Large deposits occur also in Alberta, Saskatchewan and Manitoba, Canada. A zone covering about 1700 square miles has been located in Australia, and one small deposit has been reported in England (at Bovey-Tracey in Devonshire). In Germany, however, the lignite industry has made much more rapid advances, owing partly to the scarcity of high-grade coals, and partly to the fact that the deposits are located close to large cities,

making the cost of transportation low. The lignite is accordingly used as a fuel for steam plants, for manufacturing producer gas, and for distillation purposes to recover its valuable by-products.

The descriptions of the methods which follow are based on German practice as carried out in the following localities, viz.:

(1) Near Horrem, a short distance west of Cologne in Rhine Province;
(2) In the neighborhood of Halle on the Saale, in the Provinces of Saxony and Thuringia; and
(3) At Messel, near Darmstadt in Hessen Province.

The so-called browncoal (variety of lignite) is mined at the first two localities. It is estimated that 20,000 to 25,000 tons are briquetted daily in the Cologne mining district alone, where the beds run from 30 to 350 ft. thick, averaging 75 ft. Browncoal differs somewhat from American lignite in carrying a higher percentage of moisture (about 60 per cent instead of 25 to 50 per cent). American lignites generally resemble bituminous coal in hardness and appearance, whereas browncoal is distinctly earthy in appearance and is soft and friable enough to be dug with a spade. Moreover, browncoal contains up to 13 per cent waxy constituents which are of great value as a binder in briquetting, whereas American lignites contain less than 1.5 per cent of binder, and are unsuitable for briquetting when used alone. As mined, browncoal is soft and either unconsolidated or but slightly consolidated. The Messel deposit carries about 30 per cent clay and 45 per cent water, the organic constituents apparently being combined chemically with the clay. It is greasy in consistency, having a black color with a greenish cast. The bed covers about 240 acres in hemispherical depression, and measures 480 ft. in thickness, under a cover 13 ft. thick composed of gravel and clay.

Mining Methods. Browncoal in the Cologne and Halle regions is found in stratified beds in which the layers alternately appear lighter and darker in color. The lighter layers form a brownish-black plastic and greasy mass when freshly mined, and a yellowish to light brown pulverulent substance when dry. They are characterized by the presence of waxy constituents (soluble in carbon disulfide, benzol, etc.) The darker layers form a black plastic

mass when fresh, and a dark-brown to black earthy substance after drying. They differ from the light-colored layers, in being substantially free from waxy constituents. The two varieties are sorted during the process of mining. The light-colored product resembles the mineral pyropissite.

The lighter variety of lignite has been incorrectly termed "bituminous lignite," and the darker, "non-bituminous lignite." For purposes of differentiation, we will refer to them as "retort lignite" and "fuel lignite" respectively.* Retort lignite ranges in specific gravity from 0.9 to 1.1 and melts at ignition, whereas fuel lignite has a gravity of 1.2 to 1.4, and does not melt.

It is assumed that these two varieties of lignite, since they occur in the same deposit, result from differing conditions surrounding their formation, as for example a variation in water level. Thus if the original vegetable matter containing a large amount of waxy constituents was protected from the action of atmospheric oxygen by being surrounded with water until the transformation into lignite had been completed, then the woody tissue was more or less preserved, and fuel lignite resulted. If, however, the water receded and exposed the deposit to the action of air, then the woody tissue became partly or wholly oxidized, leaving the more resistant materials behind, and resulting in the formation of retort lignite. If the process of atmospheric oxidation had been carried to the greatest possible extent, then the waxes only remain behind, in the form of the mineral pyropissite, which, however, is no longer mined, since its total available supply has been exhausted. Lignite as freshly mined is more or less rapidly acted upon by atmospheric oxygen, the dark variety being more susceptible than the light one. A typical lignite vein carries about twice as much fuel lignite as retort lignite.

Lignite is mined by the open-cut method where the over-burden is not very thick, or by driving shafts and tunnels when the bed is situated some distance below the surface. In the case of open-cut mining, the overburden is first removed with steam shovels, and the lignite excavated by mechanically operated chain and buckets, which load the material into small skips.

* In Germany they are termed "Distillation Coal" (Schwelkohle) and "Fire Coal" (Feuerkohle).

Shaft mining presents a number of difficulties owing to the softness and unstability of the crude lignite. The shafts must be well timbered, and in many cases it is first necessary to freeze the lignite before it can be handled.

Methods of Distilling. Retort lignite is treated in one of two ways, viz.:

(1) It is subjected directly to low-temperature destructive distillation [5] or

(2) It is first extracted with a solvent to remove the montan wax and the residue either distilled destructively or briquetted and sold as fuel.

Fuel lignite is also treated in one of two ways, viz.:

(1) If it is comparatively free from ash, it is briquetted and used as fuel; or

(2) If it contains a large proportion of ash, as with Messel lignite, it is used for manufacturing producer gas by combustion in an atmosphere of air and steam, so that practically all the carbonaceous matter is consumed, leaving almost pure ash behind. Since Messel lignite in its crude state contains but 25 per cent of combustible material, it is unsuitable for use as fuel, or for purposes of destructive distillation.

When the lignite is to be used for fuel, it is converted into briquettes by subjecting the granulated material to great pressure. The heat generated during this operation softens the waxy substances present, and binds the particles into a solid mass. It is unnecessary, therefore, to add any extraneous binding medium.

In manufacturing briquettes, the lignite is first crushed to about the size of peas, then passed through a drier to reduce the moisture to approximately 15 per cent. A tubular drier, heated with steam, has been found most satisfactory for the purpose.[6] The lignite powder is fed into a briquetting press, where it is subjected to a pressure between 18,000 and 22,500 lb. per square inch.

Retort lignite is unsuitable for fuel or manufacturing briquettes, as the large quantity of waxy constituents present will soften when heated, causing the briquettes to melt and drop through the grate

bars. When the retort lignite has been extracted with solvents to remove the "montan wax," the residue retains enough waxy constituents to enable it to be briquetted.

When the retort lignite is to be subjected to destructive distillation, it is used directly as it comes from the mine, without drying. In fact, the presence of the water materially assists the distillation process by preventing the volatile products from decomposing too extensively. The water is converted into steam which quickly removes the vapors from the hot retort and prevents cracking, thus increasing the yield of tar. Taking the yield of tar from freshly mined lignite as 100, the yield from air-dried lignite is about 74, and from lignite dried at 105° C. approximately 56.

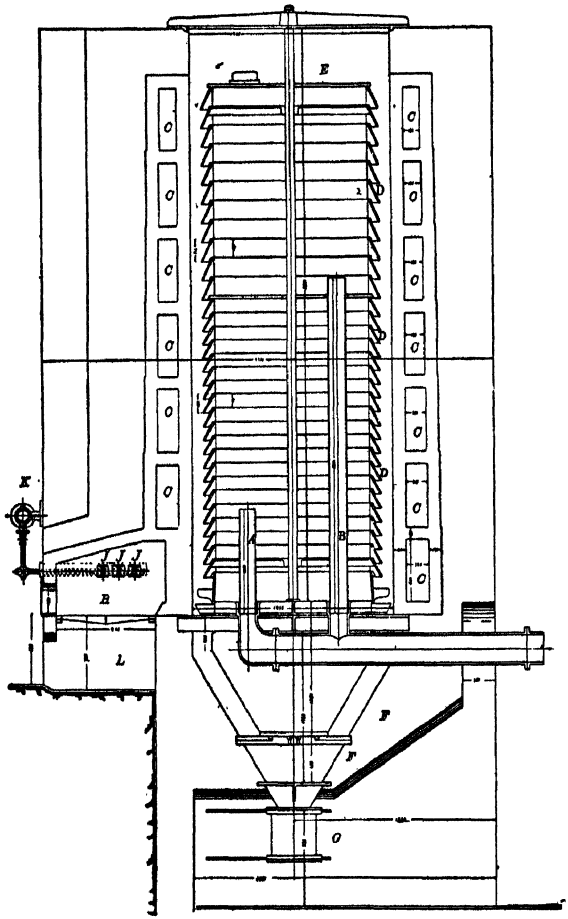


FIG. 96.—Retort for Distilling Lignite.

Practice has shown that the moisture content should not be less than 30 per cent. In distilling lignite, the humic acids present are converted into the so-called "neutral bodies," the cellulose derivatives into phenolic bodies and unsaturated hydrocarbons, and the waxy constituents into saturated hydrocarbons and paraffin wax.

It is claimed that the Rolle retort shown diagrammatically in Fig. 96 has been found most satisfactory for treating 3-5 tons lignite per day. It is 5 to 6 ft. in diameter by 20 to 25 ft. high, and works continuously, the operation progressing in two stages, viz.:

(1) Drying the lignite.
(2) Decomposing the lignite into gas, water, tar and coke.

The contrivance is composed essentially of two concentric cylinders, an outer one of fire brick and an inner one consisting of a stack of conical rings assembled in louvre fashion, constructed of iron or fire clay. The lignite after being crushed into lumps about 1½ to 2½ in. in diameter is introduced into the space between the concentric cylinders. The products of distillation pass out through the flues *A* and *B*. The openings *C* represent the fire-flues; *D*, the stack of conical rings; *E*, the cap covering the rings; *F*, an inverted cone of metal into which the coke falls after the lignite has been thoroughly carbonized; *G*, a device for intermittently drawing off the coke; *H*, the combustion chamber; *J*, vents for introducing the gases; *K*, the pipes through which the gases enter; and *L*, the fire place which comes into play when the retort is first started up. Coal or lignite is burnt on the grate, until the process of destructive distillation commences, whereupon the resulting non-condensable gases are introduced through *K* and *J*, and caused to burn in the flues *C*. The space over the cap *E* is kept filled with lignite, and the rate of travel through the retort is controlled by the frequency with which the coke is removed from the chamber *G*.

The temperature at which the distillation takes place varies between 500 and 900° F., and the vapors issue from the retort at 250 to 300° F. The products of decomposition are drawn from the retort by a slight suction, and passed through a series of air condensers, which removes most of the tar, the high boiling-point oils, and part of the water. The condensation is completed by passing the gases through pipes surrounded by water.

The tar is separated from the condensed water by warming it and allowing it to stand quietly in a suitable receptacle. The tar being lighter than the water, rises to the surface, and is drawn off when the separation is complete.

Products Obtained. A bituminous lignite containing 20-30 per cent moisture and 15-16 per cent soluble in carbon disulfide, yielded 24 per cent tar having a specific gravity of 0.885 at 50° C., a solidifying point of 37° C., and containing 16 per cent creosote oils. Similarly, a non-bituminous lignite containing 15 per cent

moisture and 3-4 per cent soluble in carbon disulfide, yielded 7.6 per cent tar having a specific gravity of 0.955 at 50° C., a solidifying point 33° C., and containing 37 per cent creosote oils. On fractional distillation with superheated steam, the former yielded 3.2 per cent pitch and the latter 8.5 per cent.

The following yields are obtained:

Lignite tar (vertical retorts)	up to 7 per cent
" " (low temperature)	7 to 12 per cent
Brown-coal tar (Rolle retort)	4 to 8 per cent
" " " (low-temperature)	8 to 24 per cent

A commercial product known as "kaumazite" is made from Bohemian lignite by a process of low-temperature distillation.

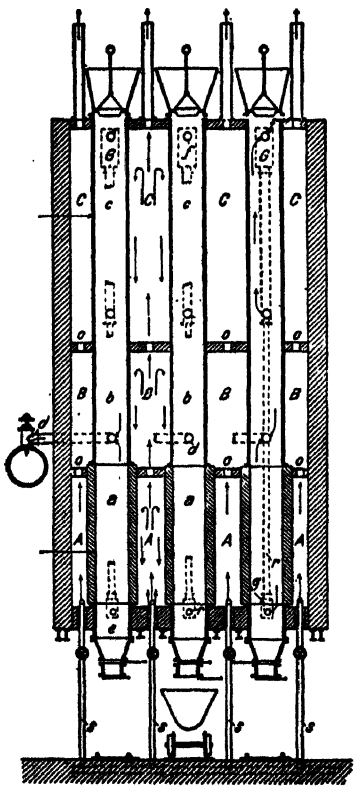


FIG. 97.—Retort for Distilling Impure Lignite.

In recent years the following products have been recovered:

Water	50–60 per cent
Tar	5–10 per cent
Coke	25–35 per cent
Gas	Balance

Treating Impure Lignite. The Messel lignite carrying a large percentage of mineral matter is treated in a special form of retort built in batteries, as illustrated in Fig. 97. The process takes place in three stages, viz.:

(1) Drying of the lignite and accompanying generation of steam, taking place in the zones *c*.

(2) Distillation of the dried material, taking place in zones *b*.

(3) Combustion of the residual coke by means of air and the steam generated in (1), taking place in zones *a*. The steam liberated in zones *c* is passed through the flues *G-e*, *f-f*, and *G-g*, respectively, into zones *a*, as illustrated.

In other words, the steam generated by the lignite itself, is used to decompose the coke into producer gas. The gas is caused to burn

in the chambers *A*, *B* and *C* respectively, the products of combustion passing through the openings *o*, *o*. Pipe *d* represents the outlet for the products of decomposition, and *s* represents the supply pipe for the heated gas. The paths of the products are indicated by the arrows. The yield of tar varies between 4 and 14 per cent, averaging about 7½ per cent (19 gallons per ton), that of gas 6 per cent, water 44 per cent and coke 36 per cent. The residue discharged from the bottom of the retort is composed of mineral matter carrying 8 per cent of undecomposed carbon. More gas is generated during the process than is required for heating the retort, hence the excess is used for other purposes.

Lignite in either the air-dried or briquetted form is gradually being used more and more, especially in Europe, for manufacturing producer gas. About 60 cu. ft. of gas are produced from each pound of the dry lignite, also ¼ to ½ per cent by weight of lignite tar, which is separated from the producer gas in the usual manner.

In certain localities (e. g. northern Bohemia) lignite is carbonized in a variety of coke-oven, similar to that used for treating coal, to produce coke, during which process a tar is obtained known as coke-oven lignite-tar. The carbonization takes place at 1100° C. and the resulting tar has a specific gravity at 60° C. of 0.970.

Properties of Lignite Tar. Lignite tar has a buttery consistency at ordinary temperatures and a dark brown to black color. It is composed of liquid and solid members of the paraffin and olefine series, together with a small quantity of the benzol series, also the higher phenols and their derivatives (10 to 25 per cent). It is characterized by the presence of a substantial proportion of solid paraffin (10 to 25 per cent) and from 0.5 to 1.5 per cent of sulfur.

Four different varieties of lignite tar are recognized, viz.:

(1) Retort tar (e. g. from Rolle retorts).
(2) Low-temperature lignite-tar (produced below 600° C.).
(3) Producer-gas lignite-tar (e. g. from Messel lignite).
(4) Coke-oven lignite-tar (e. g. northern Bohemia).

Lignite tars are usually emulsified with water, which separates with difficulty, and when derived from gas-producers they carry a certain amount of free carbon (up to 3 per cent). Both may be removed by centrifuging. Thus a tar carrying 11.7 per cent water and 2.28 per cent free carbon, upon centrifuging at 4000 to 6000

r.p.m. at 60-85° C., yielded a product carrying 0.7 per cent water and 0.06 per cent free carbon.

In general, dehydrated lignite tar conforms with the following characteristics:

(Test 1)	Color in mass	Yellowish brown to greenish brown to brownish black
(Test 7)	Specific gravity at 77° F.	0.85–1.05
(Test 9)	Hardness or consistency at 77° F.	Salve-like to buttery
(Test 10)	Ductility at 77° F.	None
(Test 15*a*)	Fusing-point (K. and S. method)	60–90° F.
(Test 15*b*)	Fusing-point (R. and B. method)	75–100° F.
(Test 16)	Volatile matter at 500° F., 5 hrs.	70–85 per cent
(Test 17*a*)	Flash-point (Pensky-Martens tester)	75–90° F.
(Test 19)	Fixed carbon	5–20 per cent
(Test 20*a*)	Distillation test	The boiling-point ranges between 80 and 400° C., the greater portion distilling between 250 and 350° C.
(Test 21)	Soluble in carbon disulphide	96–100 per cent
	Non-mineral matter insoluble	0– 2 per cent
	Mineral matter	0– 1 per cent
(Test 22)	Carbenes	0– 2 per cent
(Test 23)	Solubility in 88° petroleum naphtha	98–100 per cent
(Test 28)	Sulfur	0.5–2.5 per cent
(Test 29)	Nitrogen	Less than 0.1 per cent
(Test 30)	Oxygen	5–10 per cent
(Test 31)	Free carbon	0–1 per cent
(Test 32)	Naphthalene	½–1 per cent
(Test 33)	Solid paraffins	10–25 per cent
(Test 34*b*)	Sulfonation residue	10–20 per cent
(Test 37*e*)	Saponifiable constituents	5–20 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	No

Marcusson and Picard[7] have reported the following percentages of saponifiable constituents present in normal tar from Saxon-Thuringian lignite: saponifiable constituents 7.4 per cent, consisting of oxy-acids soluble in ether 2.4 per cent; reported as fatty acids 3.0 per cent; and phenols 2.0 per cent. Generator tar from Saxon-Thuringian lignite: saponifiable constituents 14.0 per cent, composed of oxy-acids soluble in ether 0.5 per cent; oxy-acids insoluble in ether 4.0 per cent; reported as fatty acids 4.5 per cent; and phenols 5.0 per cent.

Refining Processes. In practice, lignite tar is distilled to separate various oils and paraffin wax. The distillates are purified by

treatment with acids and alkali, and the paraffin by re-crystallization.

The distillation is conducted in one of three ways, viz.:

(1) At atmospheric pressure, without steam.
(2) By means of steam, sometimes after treating with alkalies.[8]
(3) Under vacuum, sometimes supplemented with steam.

Vacuum distillation is generally used, as it saves fuel, reduces the time and prevents cracking of the distillates. The best practice consists in using a slight vacuum at the beginning of the distillation, and gradually increasing it until the paraffin begins to distil, when it is maintained at 16 to 28 in. of mercury by a steam injector, or vacuum pump.

With steam distillation, either plain or superheated steam may be used and direct heating of the retort may be dispensed with in the latter case.

The distillation may be intermittent or continuous. European practice provides for the continuous distillation of the dehydrated tar in a battery of vertical cylindrical stills with hemispherical bottoms, having dome-shaped tops. Each still is connected with a condenser composed of a circular coil of metal piping immersed in a water tank. Between 10 and 20 stills are erected side by side on a common brick setting.

Lignite tar is first distilled to ¼ its original bulk, and the combined residues of several stills are run into a separate retort. In some cases the residues are distilled to produce lignite-tar pitch, but in the majority they are distilled until nothing but coke remains. By thus treating the residues in separate retorts, the lives of the first retorts are lengthened materially, and the wear and tear concentrated on a few. The retorts in which the preliminary distillation takes place are of course subjected to a much lower temperature than those in which the residues are treated.

When lignite tar is distilled to coke, a certain amount of cracking occurs and consequent formation of tarry matter in the distillates, which is removed by treating with sulfuric acid. The resulting sludge is worked up into lignite-tar pitch as will be described later.

Obviously the pitches derived in these two ways differ in their

physical properties, and particularly in the quantity of associated paraffin, which is smaller in lignite-tar-sludge pitch.

Products Obtained. The tar is fractioned into crude oil (about 33 per cent), a paraffinaceous distillate (about 60 per cent), red oil (about 3 per cent), and yields permanent gases (about 2 per cent), and coke (about 2 per cent).

The crude oil is re-distilled into naphtha, illuminating oil, cleaning oil, gas oil and light paraffin oil (vaseline oil). The paraffinaceous mass is cooled and pressed, which removes the heavy paraffin oil from the paraffin wax. The paraffin wax is then re-crystallized and separated into the soft paraffin wax and hard paraffin wax respectively.

According to Waldemar Scheithauer, an average grade of lignite tar will yield the following products, viz.: benzine 5 per cent, lubricating oil 5 to 10 per cent, light paraffin oils 10 per cent, heavy paraffin oils 30 to 50 per cent, hard paraffin 10 to 15 per cent, soft paraffin 3 to 6 per cent, dark-colored products 3 to 5 per cent, coke, gas and water 20 to 30 per cent. If the distillation of lignite tar is not continued to coke, lignite-tar pitch is obtained, amounting to about 5 per cent by weight of the tar.

The diagram in Table XXI shows the essential steps in treating lignite tar by fractional distillation, including the two alternatives of running to pitch and coke respectively.

The fractions are purified by treating successively with weak and strong sulfuric acid, followed by caustic soda, which improve the color and odor, and enable the products to command a higher price. The preliminary treatment with weak sulfuric acid removes a portion of the basic constituents, including the pyridine. The stronger sulfuric acid extracts the remaining basic substances, the tarry matters which impart a dark color, a portion of the unsaturated hydrocarbons and the resinous constituents. The alkali serves to neutralize the acid, and to remove the creosote oils which would impart a disagreeable odor and darken on exposure to light.

After the chemical treatment, the acid and soda sludges are settled off. The acid sludge is boiled with steam in lead-lined vessels, which decomposes it into pitch and sulfuric acid (30 to 40° Baumé). This acid is used for decomposing the soda sludge into creosote oil and sodium sulfate (Glauber salt). The impure creo-

TABLE XXI

LIGNITE TAR
Distilled Destructively
Lignite-tar Pitch (from direct distillation)
1
2
Coke
Gases (Burned under retorts)
Red Oil
Paraffinaceous mass (cooled and pressed)
Paraffin Wax (recrystallized)
Hard Paraffin Wax
Soft Paraffin Wax
Heavy Paraffin Oil
Crude oil (redistilled)
Naphtha
Illuminating Oil
Cleaning Oil
Gas Oil
Light Paraffin Oil
Refined by treating with sulfuric acid and caustic soda respectively
Soda Sludge
Acid Sludge (boiled with steam)
Weak sulfuric acid
Acid Sludge Pitch
Mixed together
Impure Creosote Oil
Sodium Sulfate
Mixed together
Washed and steam-distilled
Refined Creosote Oil
Lignite-tar Pitch (from acid sludge)

sote containing tarry matters is mixed with the pitch separated from the acid sludge, and after washing with water to remove all traces of acid and alkali, the mixture is distilled with superheated steam. The purified lignite creosote is recovered as distillate (having a specific gravity of 0.940 to 0.980, and yielding 50 to 70 per cent soluble in caustic soda) and the lignite-tar pitch remains as residue. The extent to which the distillation is continued regulates the hardness and fusing-point of the pitch, which is much harder in consistency than that obtained from the direct distillation of lignite tar. A black, glossy pitch of good quality may be produced by treating lignite tar with 4 per cent sulfuric acid (60° Baumé) at 160° C. and the lower pitch-like layer heated with 20 per cent anthracene oil at 250° C.[9]

Properties of Lignite-tar Pitch. Lignite-tar pitches conform with the following tests:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	Uniform
(Test 3)	Appearance surface aged indoors one week	Dull
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Very bright when fresh
(Test 6)	Streak	Black
(Test 7)	Specific gravity at 77° F.	1.05–1.20
(Test 9*b*)	Penetration at 77° F.	0–60
(Test 9*c*)	Hardness at 77° F., consistometer	10–100
(Test 9*d*)	Susceptibility index	Greater than 100
(Test 10)	Ductility	Variable
(Test 15*a*)	Fusing-point (K. and S. method)	90–250° F.
(Test 15*b*)	Fusing-point (R. and B. method)	100–275° F.
(Test 16)	Volatile matter	Variable
(Test 17*a*)	Flash-point	Usually above 250° F.
(Test 19)	Fixed carbon	10–40 per cent
(Test 21)	Solubility in carbon disulfide	95–99 per cent
	Non-mineral matter insoluble	0– 2 per cent
	Mineral matter	0– 1 per cent
(Test 22)	Carbenes	0– 5 per cent
(Test 23)	Solubility in 88° petroleum naphtha	60–85 per cent
(Test 25)	Solubility in benzol	75–90 per cent
(Test 28)	Sulfur	1.5–2.5 per cent
(Test 30)	Oxygen in non-mineral matter	2– 5 per cent
(Test 31)	Free carbon	Trace
(Test 32)	Naphthalene	Absent
(Test 33)	Solid paraffins	1– 5 per cent
(Test 34*b*)	Sulfonation residue	5–15 per cent
(Test 37*e*)	Saponifiable constituents	0– 8 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	No

The following figures are reported by Julius Marcusson:[10]

		Straight-distilled Lignite-tar Pitch	Lignite-tar Pitch Derived from Acid-sludge
(Test 9)	Hardness	Moderately hard	Hard and brittle
(Test 15*a*)	Fusing-point (K. and S. method)	88° F.	190½° F.
(Test 24)	Free carbon	11 per cent	20 per cent
(Test 28)	Sulfur	< 2 per cent	> 2 per cent
(Test 37*a*)	Acid value	0.5	4.4
(Test 37*d*)	Saponification value	2.8	13.4
(Test 37*e*)	Saponifiable constituents	1.8 per cent	6.0 per cent
(Test 38*c*)	Asphaltenes	24 per cent	36 per cent
(Test 38*d*)	Asphaltic resins	11 per cent	8 per cent
(Test 38*e*)	Oily constituents*	48 per cent	26 per cent

* Salve-like; light brown color.

Soft lignite-tar pitch is almost completely soluble in benzol and turpentine, and less soluble in petroleum benzine or naphtha. The solubility of hard pitches decreases in proportion to the extent they have been distilled. Lignite-tar pitch derived from acid sludge may be distinguished from straight-distilled pitches by the presence of sulfuric-acid oxonium derivatives insoluble in 88° petroleum naphtha, as is the case with sludge asphalts derived from petroleum. Lignite-tar pitches are distinguished from coal-tar pitches by the absence of anthracene, the presence of small quantities of paraffin wax, and the formation of water-insoluble sulfo-derivatives upon treatment with concentrated sulfuric acid (whereas coal-tar pitches form soluble derivatives). Furthermore, the free-carbon separated from lignite-tar pitches differs from that obtained from coal-tar pitches by the fact that the former is completely converted into soluble nitro-derivatives upon heating with fuming nitric acid. Lignite-tar pitches are distingished from wood-tar pitches by the associated sulfur and paraffin wax; also from asphalts, rosin pitch and fatty-acid pitch by the diazo reaction.[11]

A process of blowing lignite tar with air in the presence of mineral substances has been patented.[12] It has also been proposed to blow a mixture of lignite-tar pitch and refined Trinidad asphalt.[13] Lignite tars may be hardened by heating with sulfur,[14] or by heating with spent iron oxide containing sulfur (obtained in the purification of coal gas) in the presence of $FeCl_3$, $MnSO_4$, or the like.[15] In Germany, where practically all of the lignite-tar pitch is produced, it is used extensively for manufacturing cheap paints.

CHAPTER XVI

SHALE TAR AND SHALE-TAR PITCH

Shale Mining. Scotland is the home of the "shale oil" industry. The present chapter is based on the operations practiced in Scotland, since in other countries the treatment of shales has not been developed to what may be regarded as an industry scale. In the United States, for example, the distillation of shales is more or less in a process of development, and at the present writing is still in an experimental stage.

At the present time, Scotch refineries are located at Pumpherston, Broxburn, Oakbank, Addiewell and Uphall; crude-oil works at Addiewell, Broxburn, Dalmeny, Deans, Hopetoun, Niddry Castle, Oakbank, Philipstoun, Pumpherston, Roman Camp, Seafield, Tarbrax and Uphall; and candle works at Addiewell and Broxburn.

Shale is mined in the same manner as bituminous coal, by driving shafts, and then extending drifts radially. Considerable timbering is necessary, on account of the softness of the shale. When the seams are over 4 ft. in thickness, they are mined by the "pillar-and-stall" method, and when less than 4 ft. thick, by the "longwall" method.

The mineral as mined is hauled to the surface by power, and then run through a breaker, where the masses are broken into lumps measuring 4 to 6 in. in diameter. The breakers consist of a number of toothed iron discs mounted on two shafts revolving in opposite directions. The shale, upon being crushed to the proper size, is next conveyed up an incline to the top of the retort.

Retorts Used for Distillation. The retorts used have been modified from time to time to increase their efficiency, add to their durability, hasten the speed of treatment, or to improve the quality of the output. In all instances the distillation takes place in the upper part of the retort where the shale is heated to 900° F. It is then subjected to a higher temperature (1300 to 1800° F.) in the lower portion, which is in reality a gas producer, steam and air being

admitted to convert the carbonaceous residue into carbon dioxide and carbon monoxide. This generates sufficient heat to effect the distillation in the upper portion of the retort. The admission of air is carefully regulated to maintain the required temperature, without causing excessive combustion of the by-products. The steam serves to convert the nitrogen into ammonia, increases the value of the shale tar, dilutes the gas vapors, increases the velocity of the discharge, reduces the secondary decomposition of the vapors, increases the yield of paraffin products, and equalizes the temperature.

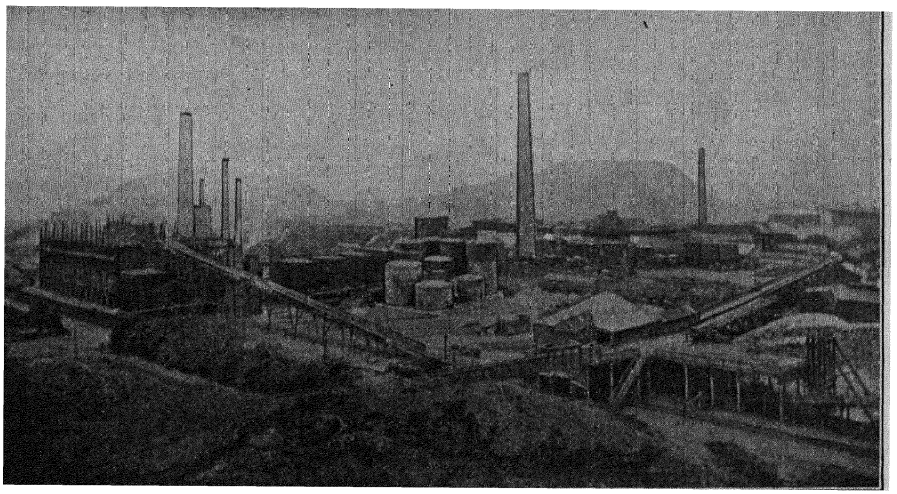


FIG. 98.—View of Pumpherston Works, Showing Mine-head, Retorts and Refinery.

The charge gradually passes downward in the retort at a speed regulated by the removal of the spent shale at the bottom.

At present, the tendency seems to gravitate towards the use of two types of retorts, viz.: the Pumpherston and the Henderson retorts, of which the former is considered to be the more efficient. Both types will now be described.

Pumpherston Retort. A view of the Pumpherston works, showing the retorts and refinery is given in Fig. 98. A bank of the retorts is illustrated in Fig. 99, and the equipment for condensing the oils and ammoniacal liquors from the gases emanating from the retorts is shown in Fig. 100. Similarly, a cross-section of a Pumpherston retort is given in Fig. 101. The shale is prevented from

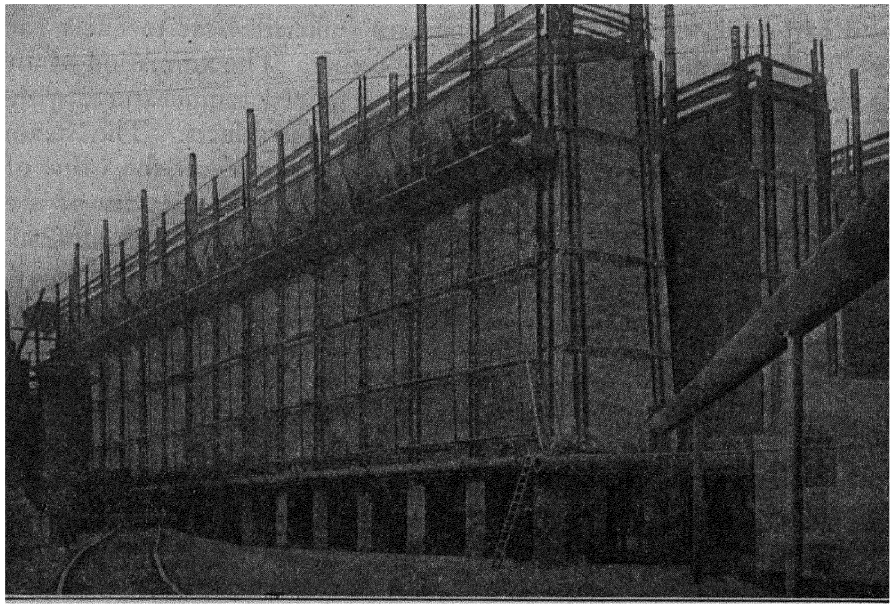


FIG. 99.—Bank of Scottish Shale Retorts (Pumpherston Type).

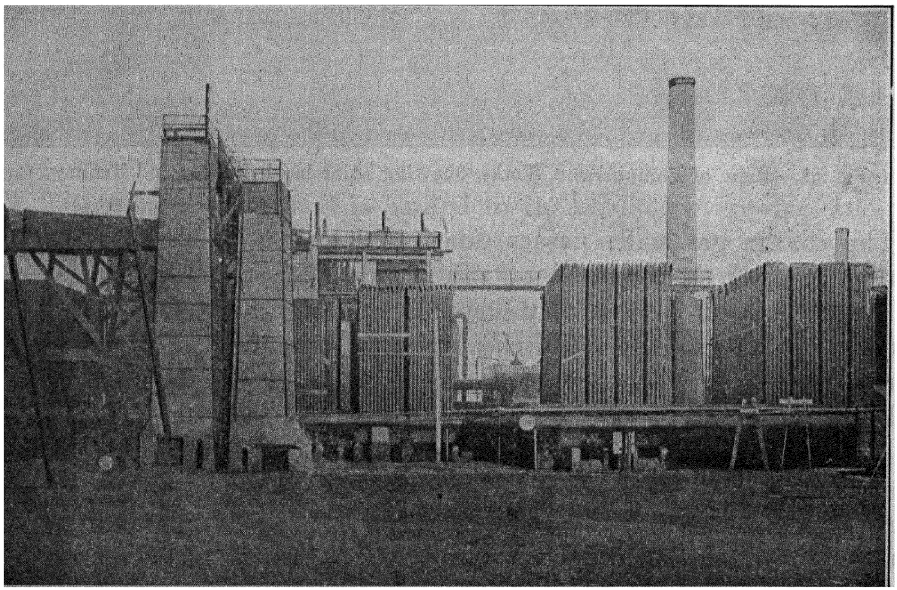


FIG. 100.—Plant for Condensing Shale Tar and Ammonia Water Emanating from the Shale.

fluxing and choking up the retort by keeping it moving continuously, which is accomplished by supporting the column of shale on a disc *e*, from which a revolving scraper *i* discharges the spent shale into the hopper below. The shale is introduced into an iron-charging hopper *c*, whence it passes into the upper cylindrical cast-iron portion of the retort *a*, measuring 11 ft. high, 2 ft. in diameter at the top and 2 ft. 4 in. at the bottom. In this, the actual distillation takes place at 900° F., with the formation of oil and gas. The shale slowly works its way into the lower portion *b* constructed of firebrick, of circular cross-section, measuring 20 ft. in height and enlarged at the bottom to 3 ft. in diameter, and finally into the lower hopper *d* extending underneath several retorts, converging in such a manner that a single line of rails running below the center will permit the spent shale to discharge into small cars. Steam is introduced into the lower portion of the retort, a short distance above the disc *e*, and the gaseous products of distillation are burned with air in the external flues at 1800° F. in the zone *b* where the ammonia is formed. The daily capacity of the retort is 4 to 4¼ tons, depending upon the richness of the shale. Four retorts constitute an

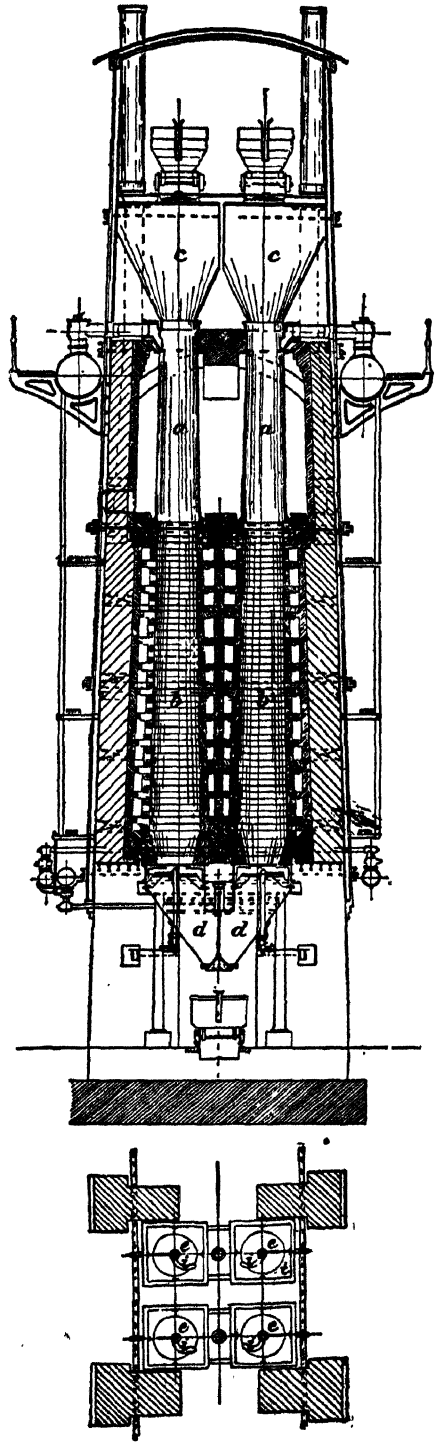


FIG. 101.—Pumpherston Type of Retort for Distilling Shale.

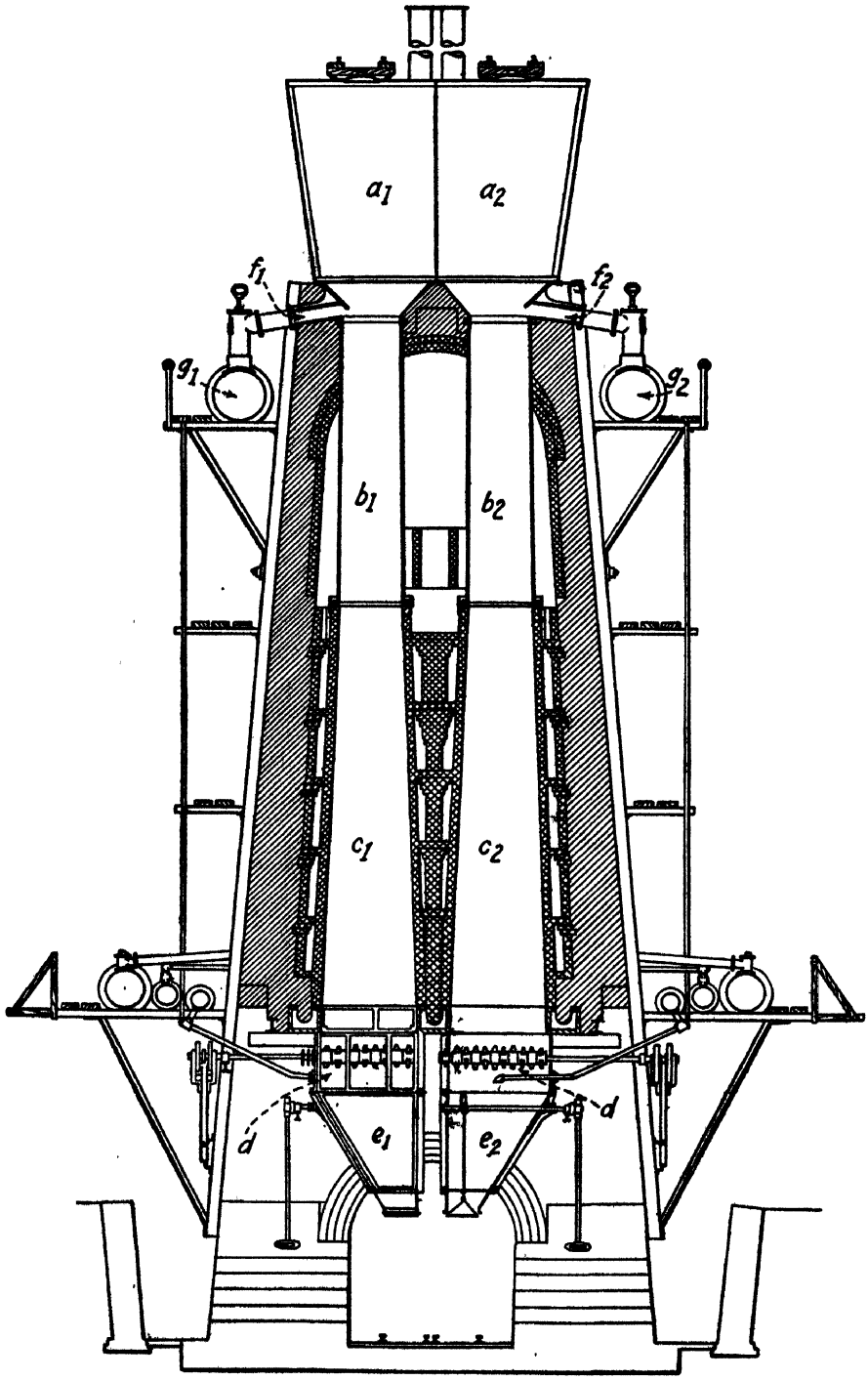


FIG. 102.—Broxburn Type of Retort for Distilling Shale.

"oven," and 16 ovens a "bench." The maximum output of shale oil amounts to 96 gal. per retort per day under the best operating conditions.

Henderson or Broxburn Retort. This is illustrated in Fig. 102, and consists of iron hoppers *a* on top, into which the crushed shale is fed, next a rectangular cast-iron section *b* measuring 2 ft. 9½ in. by 1 ft. 2⅜ in. at the top, and 3 ft. ¼ in. by 1 ft. 5¼ in. at the bottom. This is 14 ft. long, and is joined by a fire-clay joint to a fire-brick section *c*, 20 ft. long, of rectangular cross-section, measuring 4 ft. 8 in. by 1 ft. 10 in. at the bottom. A pair of toothed iron rollers *d* continuously discharge the spent shale from the bottom into hoppers *e*, which are periodically emptied into cars underneath. The vapors pass out at the top through the ducts *f*, into headers *g*. A slight vacuum is maintained in the retort and steam is admitted into the bottom of the fire-brick portion. The temperature reaches 900° F. in the upper cast-iron section, where practically all the oil is distilled, and the shale is subjected to an increasing temperature, reaching a maximum of 1500° F. at the lower portion of the fire-brick section, whereupon it is cooled by the incoming steam before it is discharged. The retorts are heated by the fixed gases evolved from the shale, supplanted by a proportion of producer gas. The arrangement of the retorts in benches and ovens, also the output, is the same for the Pumpherston type.[1]

Methods of Recovering Shale Tar. The vapors which leave the retorts are passed through air-cooled pipes (Fig. 100) which separate most of the tar and ammoniacal liquor. Sometimes an economizer is used, consisting of a tower filler with pipes, around which cold water is circulated, and thus preheated for use in the steam plant. The vapors are next passed through a scrubber (filled with coke or a checker-work of wood), and finally through a naphtha scrubber where they are washed with the "intermediate oil" obtained in distilling the shale tar (having a high boiling-point and a specific gravity of 0.84 to 0.86) which extracts any light naphtha not previously condensed (about 2 gal. per ton of shale). The naphtha is separated from the scrubbing oil by heating the mixture moderately in a still, and condensing the distillate (having a specific gravity of 0.73).

The crude tar and ammoniacal liquor are allowed to flow from the condensers to separating tanks, where upon standing, the tar rises to the surface and is drawn off and piped to the refinery. The crude tar is generally termed "shale oil," but this name is just as inappropriate as the expression "oil shale," often used to designate the shale.

Products Obtained. Upon destructively distilling the Kimmeridge Shales of England and the Lothian Shales of Scotland, the following products are obtained:

(1) Non-condensable gases, averaging 9800 cu. ft. per ton (2000 lb.).
(2) Ammoniacal liquor yielding an average of 40 lb. ammonium sulfate per ton.
(3) Shale tar, averaging 22 gal. per ton.
(4) Scrubber naphtha, averaging 0.4 gal. per ton.
(5) Spent shale, averaging between 75 and 85 per cent of the raw shale (i.e., 1500 to 1600 lb. per ton), and containing approximately 2½ per cent unconsumed carbon.

The non-condensable gases are burned under the retorts, and the spent shale discarded, as it has no further value. The valuable products are the light naphtha, the shale tar and ammonium sulfate.

The ammoniacal liquor separated from the tar is treated with steam under a pressure of 20 to 30 lb. in a tower filled with baffle-plates. The liquor is run in at the top and the steam introduced at the bottom. The ammonia is expelled in the gaseous state and recovered by passing it into sulfuric acid contained in a vessel known as a "cracker box." The acid used for this purpose is usually the waste product from the refining process. Crystals of ammonium sulfate separate when the liquor becomes sufficiently concentrated, and after being dried are marketed as such. In this manner the ammonia is separated from the other nitrogenous bases, including pyridine, contained in the aqueous liquor.

The following table gives the minimum and the maximum yields of dehydrated shale tar in gallons, and ammonium sulfate in pounds per ton of shale, obtained from the most important shale deposits in different parts of the world.[2]

	Yield of Shale Tar (Gallons)	Yield of Ammonium Sulfate (Pounds)
Lothian shale (Scotland)	10–55	6–70
Kimmeridge shale (England)	10–40	10–50
Coorongitic shale (New South Wales)	14–150	20–30
Orepuki shale (New Zealand)	20–40	.....
Albert shale (New Brunswick)	30–51	67–111
Arcadian shale (Nova Scotia)	4–23	9–40
Shales (Eastern United States)	4–45	0–10
Utah shales	6–10	40–50
Colorado and Wyoming shales	10–68	22–34
Kukkersite (Esthonia)	70–80	5–15

Properties of Shale Tar. Shale tar usually appears black in mass with a greenish fluorescence. It is similar in composition to lignite tar, although differing from the latter in containing a larger percentage of nitrogen (1.1 to 1.5 per cent). Members of the paraffin and olefine series constitute 80 to 90 per cent by weight of the tar, and small quantities of cresols and phenols are present.

Dehydrated shale tar tests as follows:

(Test 1)	Color in mass	Brownish black with a greenish fluorescence
(Test 7)	Specific gravity at 77° F.	0.85–0.95
(Test 9)	Hardness or consistency	Salve-like to buttery
(Test 15*a*)	Fusing-point (K. and S. method)	60–90° F.
(Test 15*b*)	Fusing-point (R. and B. method)	75–115° F.
(Test 16)	Volatile matter at 500° F., 5 hrs.	80–90 per cent
(Test 17*a*)	Flash-point (Pensky-Martens tester)	20–60° F.
(Test 19)	Fixed carbon	5–10 per cent
(Test 21)	Soluble in carbon disulfide	98–100 per cent
	Non-mineral matter insoluble	0–2 per cent
	Mineral matter	0–1 per cent
(Test 22)	Carbenes	0–2 per cent
(Test 23)	Soluble in 88° petroleum naphtha	95–100 per cent
(Test 24)	Free carbon	0–2 per cent
(Test 28)	Sulfur	1.5–2.5 per cent
(Test 29)	Nitrogen	0.25–1.0 per cent
(Test 30)	Oxygen	1–5 per cent
(Test 33)	Solid paraffins	5–15 per cent
(Test 34*b*)	Sulfonation residue	15–35 per cent
(Test 37*e*)	Saponifiable constituents	0–2 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	No

The percentage of phenols contained in the shale tar is very much smaller proportionately than that present in peat or lignite

tars. Shale tar is distinguished from the latter by containing larger percentages of nitrogen and sulfur, and smaller percentages of oxygen, paraffin and phenols respectively.

Refining of Shale Tar. Shale tar may be distilled either intermittently or continuously. In either case the process consists in heating the tar in a still to expel the moisture, whereupon either plain or superheated steam is introduced through a perforated pipe under a pressure of between 10 and 40 lb. The tar is distilled to coke and the following products separated:

(1) Non-condensable gases ranging from 1 to 2 cu. ft. for each gallon of shale tar.

(2) Crude naphtha having a specific gravity of 0.74 to 0.76.

(3) So-called "crude distillate," or "once-run oil," or "green oil" representing the fraction between the crude naphtha and coke.

(4) A residue of coke approximating 3 per cent by weight of the shale tar.

The steam is shut off towards the end of the distillation, after the "once-run oil" has passed over.

The stills used in Scotland are of the vertical type from 2000 to 2500 gal. capacity, constructed of a hemispherical cast-iron bottom, and a soft malleable-iron cylindrical body to which is attached a dome-shaped top bearing the exit pipe. Each still is connected with its own condenser.

In the continuous distillation process, termed the "Henderson Process," a battery of three horizontal stills and one or more vertical pot-stills are used. The tar is first led into the middle horizontal still where the naphtha is distilled off, and the residue caused to flow continuously into the two side stills. These are heated higher than the center still, causing the one-run oil to distil over continuously. The residues from these second stills are led into the pot-still, where they are evaporated to dryness, the distillate being condensed and united with the once-run oil. Several pot-stills are used, since the red-hot coke must be allowed to cool before it can be removed, which prevents this part of the process being continuous.

The once-run oil is refined by agitating it with sulfuric acid at 100° F. by compressed air. The acid sludge is run off, the oil washed with water, and then treated in another agitator with caustic soda in a similar manner.

The refined once-run oil is fractioned either by an intermittent or continuous steam distillation process, the following products being recovered:

(1) Heavy naphtha varying in gravity between 0.75 and 0.77.
(2) Illuminating oils varying in gravity between 0.78 and 0.85, and having a flash-point of 125° F.
(3) Gas-oil and fuel-oils varying in gravity between 0.85 and 0.87, and having a flash-point higher than 150° F. These are used as fuel, or for manufacturing water-gas, or enriching illuminating gas.
(4) Lubricating oils having a gravity from 0.87 to 0.91.
(5) Paraffin wax which is purified by re-crystallization or "sweating," having a fusing-point between 110 and 130° F.
(6) Still grease, which represents the distillate passing over at the close of the distillation.
(7) Still coke, which remains in the still at the close of the operation.

The various distillates, with the exception of the still grease are refined further with sulfuric acid and caustic soda, similar to the method used for treating the once-run oil. The crude paraffin wax is refined by the sweating process.

The various steps of the distillation and refining processes are illustrated in Table XXII.

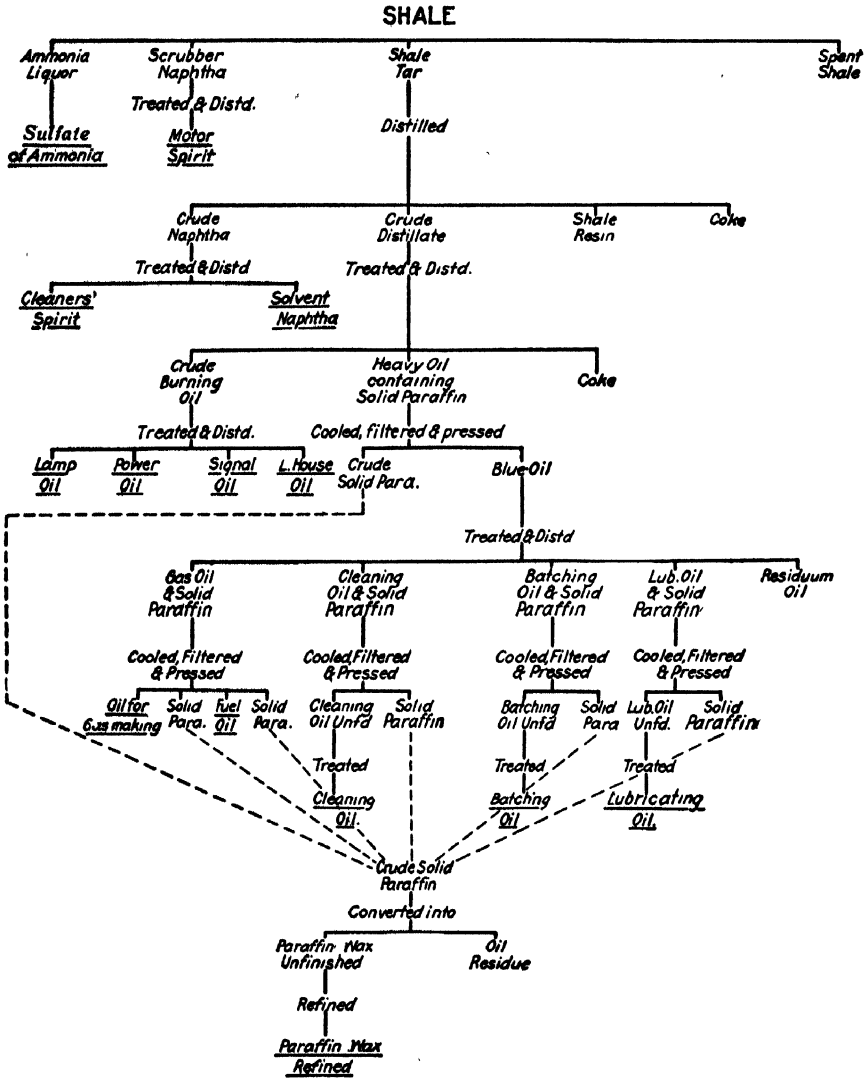
The following yields are obtained from dehydrated Scotch shale tar:

Heavy and light naphthas	3–6 per cent
Illuminating oil	20–30 per cent
Gas-oil or fuel-oil	10–25 per cent
Lubricating oil	15–20 per cent
Soft paraffin scale	3–5 per cent
Hard paraffin wax	7–9 per cent
Non-condensable gases	3–5 per cent
Acid and soda sludges and losses	20–25 per cent
Still coke	2–3 per cent

The acid and soda tars obtained from the various refining processes are mixed together in such proportions that the free acid and alkali will exactly neutralize each other. The resulting sludge is ordinarily used as fuel for the stills, but experiments have been made to convert it into pitch suitable for use as a wood preservative, pipe-dip, or the base of bituminous paints. Comparatively

TABLE XXII

FLOW SHEET ILLUSTRATING THE METHOD OF MANUFACTURE OF OILS, PARAFFIN WAX AND SULFATE OF AMMONIA FROM SHALE. THE NAMES OF FINISHED PRODUCTS ARE UNDERLINED



little has been accomplished in this direction, probably due to the fact that other products are available for these purposes, costing but little more and possessing superior weather-resisting properties. Shale-tar pitch is similar in its physical properties and composition to lignite-tar pitch.

A residue derived from Esthonian shale oil (obtained by the destructive distillation of Esthonian "kukkersite") has been termed "esto-bitumen" or "esto-asphalt," which unlike paraffin petroleum asphalts is completely miscible with coal tar in all proportions.[8]

CHAPTER XVII

COAL TAR AND COAL-TAR PITCH

Under the headings "Coal tar" and "Coal-tar pitch," will be included the tars and corresponding pitches recovered as by-products from bituminous coal [1] in:

(1) Gas works;
(2) Coke ovens;
(3) Blast furnaces;
(4) Gas producers;
(5) Low-temperature processes.

Water-gas tar and water-gas-tar pitch have been included by some writers within the scope of the terms coal tar and coal-tar pitch respectively, but in this treatise they will be considered separately, since they differ in their composition and properties, due to the use of petroleum products in their manufacture.

Bituminous Coals Used. Bituminous coals only are suitable for the production of coal tar. Cannel, bog-head and anthracite coals will not answer, since the former distils at too low a temperature, and the latter contains insufficient volatile matter. Upon subjecting cannel coal to low-temperature distillation, there is obtained 20 to 40 per cent of cannel-coal tar having a specific gravity at 50° C. of 0.9, containing much paraffin and 5 to 10 per cent phenols. At 20° C. cannel-coal tar has the consistency of butter. Upon subjecting bog-head coal to low-temperature distillation, 30 to 60 per cent of tar is produced, having a thick, salve-like consistency and containing solid paraffins, phenols and naphthenes, but no naphthalene or anthracene. It is known as bog-head-coal tar. Torbanite on distillation yields 20 to 35 per cent of torbanite tar having a specific gravity at 35° C. of 0.90 to 0.95, and containing phenols and a small amount of solid paraffin.

Bituminous coals are known as "gas coals" when used for manufacturing illuminating gas, and "coking coals" when used for coking. Western Pennsylvania, West Virginia, Virginia, eastern Kentucky

and Tennessee produce most of the bituminous coals used for these purposes, and they comply with the following characteristics:

Air-dry loss of coarse material	1 –5 per cent
Moisture at 105° C. (powdered material)	1.5–7.0 per cent
Volatile matter on ignition	20 –40 per cent
Fixed carbon	50 –75 per cent
Ash	Less than 15 per cent
Sulfur	Less than 2 per cent
Hydrogen	4.5–5.5 per cent
Carbon	65–85 per cent
Nitrogen	1–2 per cent
Oxygen	5–15 per cent

Very little is known regarding the chemical composition of the bituminous coal itself, due to the difficulty in converting the coal into recognizable derivatives, and because of its slight solubility in the usual solvents for bituminous materials. On subjecting coal to high temperatures, the bodies present decompose into simpler substances which fail to give any clue as to their original structure and composition. Recent researches lead to the conclusion that coal is essentially a conglomerate of cellulose decomposition products, admixed with altered resins and gums originally present in the plants from which the coal was derived.

Temperature of Treatment. D. T. Jones examined the tars derived from the destructive distillation of bituminous coal *in vacuo* at very low temperatures (below 450° C.). These were then subjected at atmospheric pressure to successively increased temperatures up to 800° C. Unsaturated hydrocarbons, naphthenes, paraffins, phenols, aromatic hydrocarbons and pyridines were found to be present in the low-temperature tar, whereas benzol and its homologues, naphthalene, anthracene, phenanthrene and the solid aromatic bodies were absent. As the temperature was increased, the naphthenes, paraffins, and unsaturated hydrocarbons were transformed into olefines. As the temperature was further increased, the olefines were in turn transformed into benzene and its homologues. The percentage of olefines appears to reach a maximum at 550° C., and a minimum at 750° C., at which latter temperature hydrogen and naphthalene are rapidly evolved, as well as methane. The conclusion reached is that ordinary coal tar obtained from bituminous coal at high temperatures results chiefly from the decomposition of the tar previously formed at lower temperatures.

The following figures will give a rough idea of the effect of the temperature on the yield and characteristics of the tar produced from an average grade of British coal:

	Temperature	Tar (Gals. per Ton)	Sp. Gr. Tar at 77° F.	Free Carbon in Tar, Per Cent	Distillate to 315° C.		Pitch (Per Cent by Weight)
					Naphthalene, Per Cent	Paraffin, Per Cent	
Coke-ovens (narrow)	1300° C.	8.0	1.210	20	30–35	0	72
Coke-ovens (ordinary)	1100–1250° C.	9.5	1.200	15	20–30	0	66
Horizontal retort	900–1200° C.	11.0	1.190	14	15–20	Trace	65
Inclined retort	1000–1100° C.	12.0	1.155	12	5–12	5	58
Vertical retort	1000–1200° C.	15.5	1.100	5.5	<5	13	48
Low-temperature carbonization	400–700° C.	18.3	1.035	1	0	25	40

By low-temperature carbonization of coal is meant its destructive distillation under 700 to 800° C. Below this temperature range there are evolved mostly condensable tars and oils, with a minimum of fixed gases; whereas above this range, mostly fixed gases are produced, due to secondary reactions. This is illustrated by the following figures applicable to 1 ton of average coal containing 25 to 30 per cent volatile matter:

	Low-temperature (Coalite Process)	High-temperature Processes	
		Gas Works	Coke Ovens
Temperature of vapors	550° C	1,000° C.	1,200° C.
Yield gas	6,000–6,500 cu. ft. rich gas (700–750 B.t.u.)	12,000 cu.ft. medium gas (550 B.t.u.)	11,500 cu. ft. lean gas (450 B.t.u.)
Yield liquid products	20 gal. tar (*)	10 gal. tar	8 gal. tar
Ammonium sulfate	15 lb.	25 lb.	28 lb.
Yield coke	14 cwt.	13½ cwt.	14–14½ cwt.

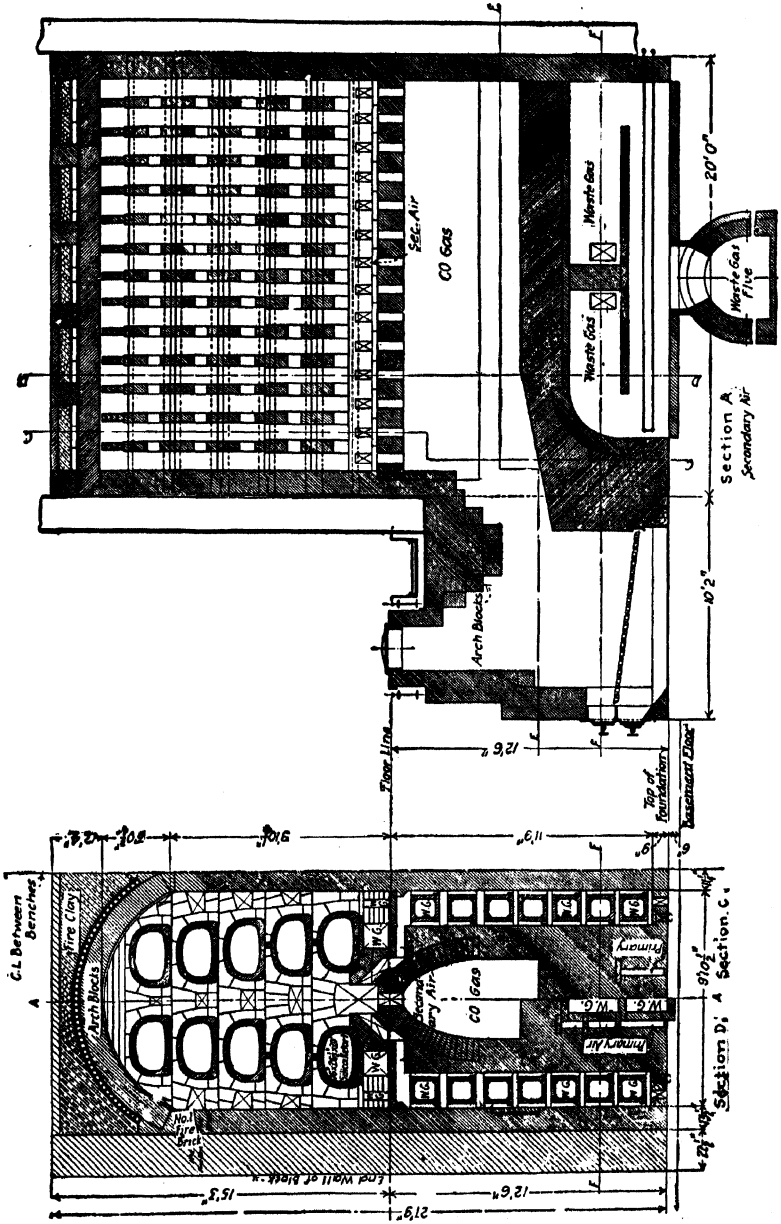
* Absence of naphthalene and anthracene distinguish this tar from high-temperature tars.

The percentage yield of tar, figured on the weight of coal treated, will approximate the following:

Gas works (horizontal retort) ..... 5–7 per cent
Gas works (vertical retort) ..... 4–6 per cent
Coke-ovens ..... 2–6 per cent
Low-temperature carbonization ..... 8–13 per cent
Blast-furnace operation ..... 5–10 per cent

The commercial processes for obtaining coal tar will now be considered.

Production of Gas-works Coal Tar. In manufacturing illuminating gas, bituminous coal is heated in comparatively small fire-



From "Coal and Coke," by F. H. Wagner.

FIG. 103.—Horizontal Gas-Works Retort.

clay retorts, of D-shaped, oval or round cross-section about 16 to 24 in. in diameter. The D-shaped retort is ordinarily used in

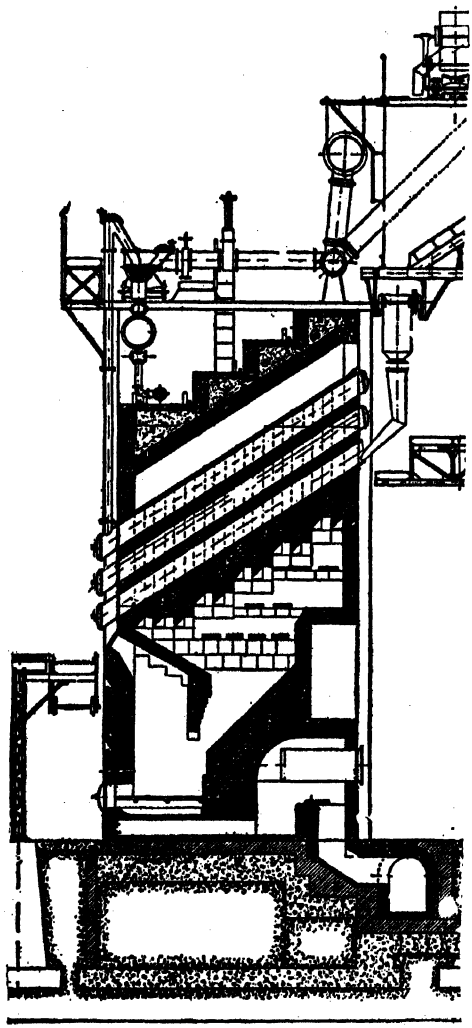
modern gas-works because it is least liable to distortion under the action of heat, and moreover presents the greatest area at its base, enabling the contents to be heated more rapidly. In some cases the retorts are "single-ended," measuring 8 to 9 ft. in length, but modern practice favors the use of "double-ended" retorts composed of three sections joined together, measuring 15 to 25 ft. over all. In the single-ended retort a metal mouthpiece is bolted to one end, to which in turn the gas outlet pipe is fastened. With the double-ended retort, metal mouth-pieces are bolted fast to both ends. From 6 to 9 retorts are set together in a common brick setting, constituting a "bench" which is heated by a single furnace.

Retorts Used. The retorts are supported in either a horizontal, inclined or vertical position. The inclined or vertical retorts seem to meet with greater favor since they avoid overheating, prevent the formation of "free carbon" in the tar, and at the same time permit the coke to be handled by gravity. The vapors leave horizontal retorts at 1600 to 1800° F. and the vertical and inclined retorts at 1300 to 1400° F.

The retorts are heated with water-gas obtained by passing air and steam through incandescent coke beneath the "bench." The coke used for this purpose is derived as a residue from a previous charge of bituminous coal, amounting to 15 to 25 per cent of the total coke produced. The water-gas is burnt in flues surrounding the retorts and the process of combustion controlled by the introduction of air. This method of firing results in a higher and more uniform temperature with the minimum consumption of fuel. The temperature in the combustion chamber ranges from 2800 to 3200° F., and in the flues surrounding the retorts from 1900 to 2200° F. An improved installation of horizontal retorts is shown in Fig. 103, inclined retorts in Fig. 104, and vertical retorts in Fig. 105. Continuously operating vertical retorts are now being adopted extensively, in which the coal is fed through the retort in a constant stream, the coke being withdrawn continuously at the bottom.

Formerly, the retorts were charged and discharged by hand, using a shovel and rake respectively. Mechanical devices are now used for the purpose, the double-ended horizontal retorts being charged at both ends with a scoop, fed from an overhead hopper, operated either by compressed air or electricity. About 600 lb. of

coal are introduced into the double-ended retort, and subjected to heat from 3 to 6 hours. The inclined and vertical retorts are charged through the top and discharged by gravity from the lower end. Horizontal retorts are discharged by a pneumatic or an electrically driven ram, which forces out the coke at the farther end. Inclined retorts are set at an angle between 25 and 35°, which is sufficient to enable the coal to feed into the lower end, where it is held in place by a metal cover. In the inclined and vertical types the volatile constituents are withdrawn from the upper end.



From "Coal and Coke," by F. H. Wagner

FIG. 104.—Inclined Gas-Works Retort.

The vapors are subjected to the highest temperatures in the horizontal retort, due to the longer contact with the heated internal surfaces, which results in a larger percentage of free carbon, and a tar of higher specific gravity.

Methods of Recovering Gas-works Coal Tar. The volatile products pass from the retort into the hydraulic main, which forms a water-seal, permitting any retort to be charged, and at the same time preventing the gas generated in the other retorts escaping through the open one. The hydraulic main reduces the temperature of the vapors to 130-160° F.

After leaving the hydraulic main the vapors are subjected to the following treatment in modern gas-works:

(1) The gases are passed through a "primary condenser" which may either be air-cooled or water-cooled, or both.

(2) The gases are then passed through a tar-extractor.

(3) Then they are passed through an exhauster to relieve the pressure on the retorts and force the gases through the ensuing train of apparatus.

(4) The gases are next passed through two "scrubbers," preferably of the rotary type. In the first scrubber the gases are washed with a heavy tar oil, such as anthracene oil, to remove the naphtha-

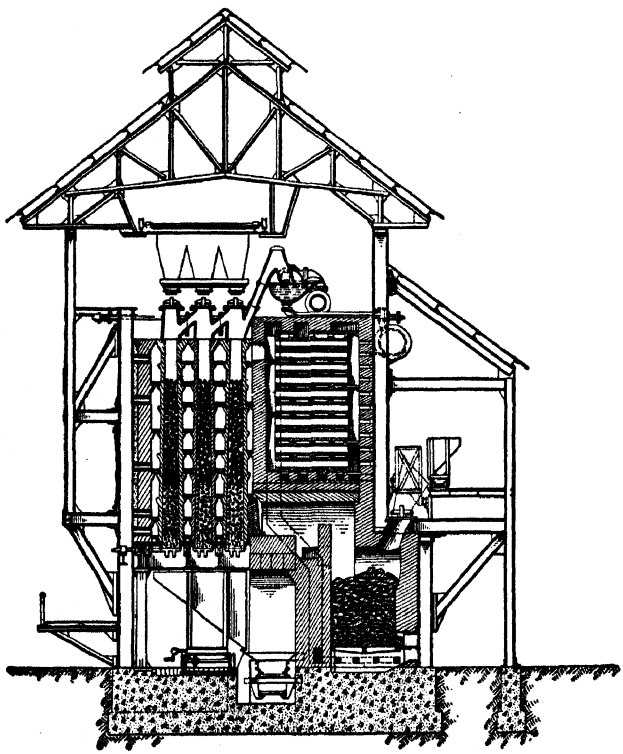


FIG. 105.—Vertical Gas-Works Retort.

lene, and in the second with an alkaline solution of ferrous sulfate to remove the cyanogen.

(5) The gases are then cooled to about 60° F. by passing them through a "secondary condenser," similar to the first one.

(6) The ammonia is next removed by passing the gases through a third scrubber through which a stream of water is allowed to trickle. Formerly a tower scrubber filled with a checker-work of wooden boards was used for this purpose, but this is being replaced by a rotary scrubber similar to that used for extracting the naphthalene and cyanogen.

(7) The last step consists in passing the gases through a series of "purifiers," consisting of low cylindrical chambers filled with trays or sieves. Some of the purifiers are filled with slaked lime to remove carbon dioxide and a portion of the sulfur compounds, and others with iron oxide to remove the remainder of the sulfur compounds (mostly hydrogen sulfide).

Products Obtained. The following percentages of tar are collected from the hydraulic main, condenser, washer and scrubber, also the tar extractor respectively:

Hydraulic main	61 per cent
Condensers	12 per cent
Washer and scrubbers	15 per cent
Tar extractor	12 per cent
Total	100 per cent

The operations which take place in the final handling of illuminating gas before it enters the mains, cease to be of interest in relation to the production of tar, and will accordingly be omitted.

In the United States, temperatures to which the retorts are heated vary from 900 to 1500° C. In England the average temperature is 1100° C. In Germany horizontal retorts are heated between 1000 and 1100° C., and inclined retorts between 1100 and 1200° C. The quantity and yield of the tar depend largely upon the temperature. In the low-temperature production of illuminating gas, an average of 16 gal. of tar is produced per ton of coal, and in high temperature processes an average of 8. The maximum variation ranges between 4 and 20 gal. of tar per ton. High-temperature processes are preferable, as they increase the yield of gas, but have the disadvantage of reducing its illuminating power.

The following represent the yields from an average grade of bituminous coal in manufacturing illuminating gas:

Gas	17 per cent (10,000 cu. ft.)
Aqueous liquor	8 per cent
Tar	5 per cent
Coke	70 per cent
Total	100 per cent

Of course, these figures are subject to variation, and depend upon the quality of bituminous coal used, the temperature at which it is distilled, etc. Thus the yield of gas per ton of rich coal will vary from 5000 to 15,000 cu. ft., and the residual coke from 55 to 75 per cent.

The tar collected from the hydraulic main, condenser, washers and scrubbers is run into wells constructed of metal or masonry, sometimes heated with steam-coils and allowed to settle as long as possible, to permit the aqueous liquor, which is lighter than the tar, to rise to the surface, where it is drawn off and treated separately to recover the ammonium compounds. The well-settled gas-works tar carries between 4 and 10 per cent of water. In exceptional cases the water may run as high as 40 per cent, although this is not regarded with favor.

Production of Coke-Oven Coal Tar.[2] As stated previously, about 78 per cent of the coal tar produced annually in the United States is obtained from coke-ovens equipped to recover by-products. This only represents between 60 and 70 per cent of the total quantity of bituminous coal converted into coke. The remaining 30 to 40 per cent is coked in brick "beehive" ovens, constructed in the form of a beehive, and not adapted to recover the gas, ammonia or tar, which are allowed to burn away through an opening in the top of the oven, thus constituting a reckless waste of our national resources, running into many millions of dollars annually. For years this wasteful practice remained unchecked, but happily the present tendency is to replace the beehive ovens with types adapted to recover by-products, and it is probably only a matter of a few years more before all the coke-ovens will be equipped to recover the gas, ammonia and tar. This same wasteful tendency is reflected in a patent granted in the United States and describing the production of coal-tar pitch by the simple expedient of igniting coal tar and permitting the more volatile constituents to burn off (sic!).[3]

In European countries, on the other hand, where the tendency has always been towards a greater economy, coke-ovens have long been perfected to recover these by-products. In this connection it must be borne in mind, whereas it is absolutely necessary to remove the tar in manufacturing coal gas for illuminating purposes, this does not prove to be the case where the coal is converted into coke for metallurgical industries. This, and the comparative cheapness of bituminous coal in the United States, also the low price commanded by the by-products until recently, will account for the laxity in conserving them.

The temperature of coking varies between 1000 and 1300° C.,

and rarely above the latter inside the retort. The external temperature of the retort may run as high as 1700° C. The adaptability of coal for coking purposes is indicated with a fair degree of certainty by the ratio of hydrogen to oxygen, together with the percentage of fixed carbon calculated on the moisture-free basis. Practically all coals with an H : O ratio of 59 per cent or over, and less than 79 per cent of fixed carbon, possess that quality of fusion and swelling necessary to good coking. Bituminous coals with a ratio down to 55 will produce a more or less satisfactory coke, whereas coals with a ratio as low as 50 are unsuitable for coking purposes.

Retorts Used. The present systems of by-product oven construction resolve themselves into two types depending upon whether the flue construction is horizontal or vertical. In either type the coking takes place in a narrow, retort-shaped chamber about 33 ft. long, from 17 to 22 in. wide, and about 6½ ft. high. The width of the chamber averages 19 ft., which has proven suitable for completing the coking within twenty-four hours. The retort holds between 12 and 14 tons of coal.

The ends of the retort are closed by means of iron doors lined with fire brick, which after being closed as tightly as possible are luted with clay to prevent the entrance of air. The coal is charged into the top of the oven, then pushed into place and leveled by mechanical devices. At the end of the coking, the doors are opened and the coke removed by a ram, the red-hot coke being immediately quenched with water.

The number of ovens in a battery varies between 40 and 100, depending upon the type of construction. The oven walls are constructed of fire brick containing about 95 per cent of silica, which on account of its very high fusing-point enables the ovens to be worked at high temperatures, and at the same time proves to be an excellent conductor of heat.

For a detailed description of the various types of coke-ovens in use, the reader is referred elsewhere.

The coking in the by-product oven is in reality a destructive distillation process, the heat required being supplied by burning a portion of the gases evolved. A large excess of gas is produced amounting to between 40 and 60 per cent of the total.

Products Obtained. The following yields per ton are recovered:

Gas	15.0–16.0 per cent (8,500–10,500 cu. ft.)
Ammonium sulfate	0.8– 1.3 per cent
Tar	3.0– 6.4 per cent
Coke	70.0–75.0 per cent

Approximately 20 per cent of the nitrogen present in the coal is converted into ammonium compounds, part of which is found in the tar as pyridine, quinoline, etc. About half of the nitrogen remains in the coke, and may be regarded as lost.

The vapors emanating from the coke-ovens are passed through various forms of condensers and scrubbers to separate the ammoniacal liquor and tar, which are similar to those used in gas works. The yield of tar recovered from coke-ovens varies from 4 to 15 gal. per ton, depending upon the kind of coal used, as well as the type of coke-oven. Over 90 per cent of the tar (anhydrous) condenses in the collector main and primary coolers, 2 per cent in the exhausters, and 7 per cent in the tax-extractor.

The following constituents have been identified in coke-oven coal tar produced in the United States:[4]

Light oil:	Per Cent
Crude benzene and toluol	0.3
Coumarone, indene, etc.	0.6
Xylenes, cumenes and isomers	1.1
Middle and heavy oils:	
Naphthalene	10.9
Unidentified oils in range of naphthalene and methylnaphthalenes	1.7
α-Monomethylnaphthalene	1.0
β-Monomethylnaphthalene	1.5
Dimethylnaphthalenes	3.4
Acenaphthene	1.4
Unidentified oils in range of acenaphthene	1.0
Fluorene	1.6
Unidentified oils in range of fluorene	1.2
Anthracene oil:	
Phenanthrene	4.0
Anthracene	1.1
Carbazol and kindred nonbasic nitrogen-containing bodies	2.3
Unidentified oils in range of anthracene	5.4
Phenol	0.7
Phenol homologues (largely cresols and xylenols)	1.5
Tar bases (mostly pyridine, picolines, lutidines, quinolines and acridine)	2.3
Yellow solids of pitch oils	0.6
Pitch greases	6.4
Resinous bodies	5.3
Pitch (460° F. fusing-point)	44.7
Total	100.0

Production of Blast-furnace Coal Tar.[5] Most blast-furnaces in the United States employ coke as fuel and a few use anthracite coal. Since all the volatile constituents have been removed from coke, and as anthracite coal contains only a very small percentage, no tar is obtained when either of these is used for smelting ores in blast-furnaces. In such cases the gases evolved are subjected to a purification process merely to remove the entrained dust, before using them for heating purposes.

Owing to the scarcity of anthracite and the high cost of bituminous coal in Europe and Great Britain, there is a tendency to reduce the operating expenses by using the latter in its raw state, without first converting it into coke. A non-coking bituminous coal must be selected for this purpose. In such cases the gases emanating from the blast-furnace carry a certain amount of tar, derived from the volatile constituents of the coal, which must be removed before they can be used for heating or power purposes. The gases also carry a comparatively large amount of dust derived from the ores in the blast furnace, of which a good portion is removed by passing the hot gases through a device known as a "dry dust-catcher."

Methods of Recovery. After being dry-cleaned, the gases are subjected to a wet-cleaning and cooling process by passing them through coolers, scrubbers, or washers. The centrifugal washer is usually preferred as it operates rapidly and economically. A part of the tar condenses in the coolers, and the balance in the scrubbers and washers. It carries a large quantity of the wash water, which may be separated.

Approximately 7 gal. of blast-furnace tar and 29 lb. of ammonium sulfate are obtained from each ton of bituminous coal fed into the blast-furnace. It appears that the iron ore and other minerals introduced with the coal, influence the yield of tar. Thus the same bituminous coal gave the following weights of tar per ton under varying conditions:

Distilled alone in gas works	214 lb. of tar
Distilled with English iron ore	66 lb. of tar
Distilled with sand	170 lb. of tar

The tar derived from blast-furnaces always carries a substantial proportion of mineral matter, which the dust-catchers fail to re-

move, and which serves to distinguish it from the other varieties of coal tar.

Blast-furnace coal tar on distillation yields a greenish-brown creosote of low viscosity, with 30–35 per cent phenolic content, also a pitch of rather paraffinoid character. A typical tar tests as follows: specific gravity at 60/60° F., 1.151; water by volume, 0.2 per cent; phenols in tar, 14.6 per cent; bases in tar, 2.5 per cent; pitch at 315° C., 56.6 per cent of medium hardness and paraffinoid character; and distillate to 315° C., 43.4 per cent, having a specific gravity at 60/60° F. of 0.980; naphthalene little to none; and mineral matter 13.0 per cent.

Production of Producer-gas Coal Tar.[6] Unless the producer-gas plants are of a large capacity (above 4000 horse-power) it does not pay to recover the by-products. The smaller producers are designed to decompose the tar vapors and convert them into permanent gases, to avoid the expense of operating a tar-separating plant on one hand, or the trouble occasioned by the tar clogging the pipes and valves on the other. In plants where the producer gas is used without separating the tar, it is necessary to shut down about one day a week to clean out the gas lines. In spite of this, it is only in tin-plate plants, where particularly clean gas is required, that the practice is followed of scrubbing the producer gas to remove the tar. Of the larger installations, the Mond by-product gas producer is virtually the only one which is designed to recover the tar and ammonium sulfate. Upon operating this type of producer at low temperatures, as high as 150 lb. of water-free tar is recovered per ton of coal, consisting of paraffins, olefines and naphthalenes, with smaller quantities of aromatic hydrocarbons and phenols.

Production of Low-temperature Tars.[7] Low-temperature carbonization processes are carried on at 400 to 700° C., or an average of 600° C., in layers about 4 in. thick. The industry dates from 1906, when Thomas Parker (who originated the term "coalite") patented a plant for carbonizing coal at low temperatures. The yields are roughly as follows per ton of coal carbonized:

(1) Gas, 3000 to 3500 cu. ft. from externally heated retorts, or 20,000 to 25,000 cu. ft. from internally heated retorts.

(2) Tar, 20 to 25 gal.

(3) Ammonium sulfate, 10 to 25 lb.
(4) Coke, 15 cwt.

Low-temperature tars are of a liquid consistency and contain a comparatively small quantity of free carbon. They consist of 50 to 80 per cent of hydrocarbons and 20 to 50 per cent of tar acids. The hydrocarbons are mainly paraffins and saturated cyclic hydrocarbons, with varying amounts of naphthenes and unsaturated hydrocarbons. Benzol, toluol, and other aromatic hydrocarbons are entirely absent, or present only in traces. The tar acids consist mostly of the higher phenols of viscous to resinous character, with very little true phenol, and comparatively little cresol or xylenol. They also contain but small quantities of nitrogen and sulfur derivatives.

The type of coal used exerts a greater influence upon the character of the tar than does the particular mode of treatment, and in general, much variation occurs in the composition and character of the tars produced commercially. The creosote oils derived from low-temperature tars are too low in specific gravity to meet the current specifications in the United States. Less pitch is obtained than with high-temperature tars, and its character is quite different, and generally poorer in quality.

The following yields are obtained from an average British medium-coking coal, subjected to various carbonizing temperatures, expressed in percentage by weight of the moisture-free coal:

	400°C.	450°C.	500°C.	550°C.	600°C.	650°C.	700°C.
Tar (per cent)	3.9	5.62	7.06	8.00	7.60	6.90	6.24
Tar (gal. per ton)	9.1	12.8	16.0	17.65	16.4	14.35	12.9
Tar (specific gravity at 60° F.)	0.958	0.980	0.986	1.015	1.039	1.078	1.080
Aqueous distillate (per cent)	5.6	7.01	7.92	7.70	8.12	8.20	7.18
Gas (per cent)	2.2	3.32	4.05	7.14	9.10	10.54	14.58
Coke (per cent)	88 2	83.75	80.50	77.00	75.00	73.00	71.00
Loss (per cent)	0.1	0.30	0.47	0.16	0.18	0.36	1.00
Pitch (per cent in tar)	22.2	28.8	27.4	33.4	34.6	41.0	52.1
Pitch (fusing-point degrees C.)	41.2	37.0	52.2	64.5	90.5	93.5	95.8
Pitch (specific gravity at 60° F.)	1.13	1.12	1.14	1.17	1.23	1.25	1.20
Pitch (free carbon, per cent)	14.2	5.3	4.6	13.0	28.1	26.8	25.1

In Germany, low-temperature tar is termed "Urteer," and in Great Britain it is known under various names, such as "coalite

tar," "carbocoal tar," "Mond tar," "Delmonte tar," etc., depending upon the particular type of apparatus in which it is produced.

The reader is referred to other sources for a complete description of the numerous types of retorts which have been devised for the low-temperature process of carbonizing coal.

Properties of Coal Tars. As stated previously, the expression "coal tar" is properly applied to tars derived directly from coal without admixture of petroleum. Coal tars differ in their physical properties, depending upon their method of production. The following types are distinguished:

(1) Gas-works coal tar
  (*a*) Horizontal retorts
  (*b*) Inclined retorts
  (*c*) Vertical retorts
(2) Coke-oven coal tar
(3) Blast-furnace coal tar
(4) Gas-producer coal tar
(5) Low-temperature coal tar.

The figures in Table XXIII will give a general idea of the physical properties of the main types of coal tar in their dehydrated state:

The following colorimetric test may be used to detect low-temperature tars: on shaking 1–2 ml. of the oily distillate with 5 ml. aqueous ferric oxalate or ferric citrate, the aqueous layer will assume a dark blue coloration, which will turn a lemon-yellow upon acidifying with N/10 sulfuric acid and then regain its blue tint on neutralization, but turns purple and then bright red upon adding an excess of alkali. High-temperature tar oils will not give this reaction.[8]

Methods of Dehydrating Coal Tar. It is customary to dehydrate coal tars at their point of production, to avoid paying freight charges on the water content. Provision is usually made for the gravity separation in storage tanks of as much water as possible, but the amount that may be removed in this manner varies in the case of different tars, their mode of production, and other factors. With high-temperature coal tars the settled product is in the nature of an emulsion, with the water as the disperse phase. The amount of water held in stable suspension is greater in the case of horizontal

TABLE XXIII. PROPERTIES OF COAL TARS

		Gas-Works Coal Tars			Coke-oven Coal Tar	Blast-Furnace Coal Tar	Gas-Producer Coal Tar	Low-temperature Coal Tar
		Horizontal Retorts	Inclined Retorts	Vertical Retorts				
(Test 1)	Color in mass	Black	Black	Black	Black	Brownish-black	Brownish-black	Brownish-black
(Test 2a)	Homogeneity to the eye at 77° F.	Gritty	Gritty	Smooth	Smooth to gritty	Smooth to gritty	Smooth to gritty	Smooth
(Test 2b)	Appearance under microscope	Lumpy	Lumpy	Sl. lumpy	Sl. lumpy	Lumpy	Lumpy	Sl. lumpy
(Test 7d)	Specific gravity at 77° F	1.18–1.33	1.15–1.25	1.08–1.16	1.15–1.26	0.95–1.10	1.12–1.20	0.95–1.12
(Test 8a)	Engler viscosity at 212° F. (100 cc)	150–650	100–300	25–50	30–110	75–100	100–∞	25–50
(Test 15c)	Fusing-point (cube method)	Below 40° F.	Below 30° F.	Below 25° F.	Below 25° F.	Below 25° F.	Below 50° F.	Below 25° F.
(Test 16b)	Distillation test:							
	Distillate to 315° C. (by vol.) (per cent)	10–35	15–45	25–55	20–45	35–55	30–50	30–80
	Sp. gr. ditto at 60°/60° C	1.02–1.04	1.02–1.04	1.00–1.01	1.02–1.05	0.94–0.98	0.95–0.97	0.95–1.00
	Tar acids in ditto (per cent)	5–20	10–25	20–30	0–12	20–30	5–15	20–50
	Residue at 315° C. (by vol.) (per cent)	60–90	55–85	40–70	55–80	45–65	50–70	20–70
(Test 19)	Fixed carbon (per cent)	15–40	15–40	15–30	14–40	10–25	10–30	5–15
(Test 21)	Soluble in carbon disulfide (per cent)	60–85	80–85	94–98	83–97	65–80	60–95	90–100
	Non-mineral matter insoluble (per cent)	16–40	15–20	2–5½	3–17	10–25	5–25	0–7
	Mineral matter (per cent)	0–½	0–½	0–½	0–½	10–15	0–25	0–1½
(Test 22)	Carbenes	0–2	0–2	0–2	0–2	0–2	0–2	0–1
(Test 23)	Soluble in 88° petroleum naphtha (per cent)	20–40	20–60	60–80	60–75	50–70	50–75	75–90
(Test 24)	Free carbon (per cent)	20–45	10–25	2–8	2–20	15–30	5–30	½–10
(Test 25a)	Water in "dehydrated" tar (per cent)	0–5	0–5	0–5	0–5	0–5	0–5	0–5
(Test 28)	Sulfur (per cent)	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1
(Test 31)	Tar acids in tar (per cent)	1–4	4–6	5–11	1–5	5–15	3–9	10–30
(Test 32)	Naphthalene (per cent)	3–8	2–5	0–4	7½–15	0–2	0–5	0–2
(Test 33)	Solid paraffins (per cent)	0–Tr.	0–2	0–5	0–Tr.	3–15	0–3	3–15
(Test 34a)	Soluble in conc. H_2SO_4 (per cent)	98–100	96–99	95–98	98–100	90–95	98–100	90–95
(Test 34b)	Sulfonation residue of distillate 235–315° C. (per cent)	0–5	5–10	5–10	0–5	15–25	0–5	5–25
(Test 37c)	Saponifiable matter (per cent)	2–5	2–5	7½–15	2–5	10–15	5–15	15–40
(Test 39)	Diazo reaction	Yes	Yes	Yes	Yes	Yes	Yes	Yes
(Test 40)	Anthraquinone reaction	Yes	Yes	Yes	Yes	No	Yes	No

gas-retort tar than in coke-oven and vertical gas-retort tar, and still less with water-gas tar, ranging from 3 to 10 per cent in the case of the first named, down to 1 to 2 per cent in the last. Sometimes, the emulsion is of a different nature, in which the tar forms the disperse phase and the water content ranges from 15 to 70 per cent. Such emulsions are most difficult to break up by the usual means of settling, even at high temperatures. They may, however, be treated by a partial distillation, as will be described later.

Dehydration may simply imply the removal of the bulk of the water from a tar, so as to render it suitable for distillation, or as is the case in Great Britain, it may include the removal of the light oils as well, resulting in the production of a refined tar suitable for road purposes or other uses.

The following methods have been used for dehydrating tars:

(1) *Settling.* When the tar is allowed to settle in storage tanks, the water rises to the surface, where it is drawn off through a series of outlet pipes in the side. This process may be facilitated, particularly in the case of viscous tars, by heating to about 200° F. by means of steam coils in the bottom of the tank, supplied with exhaust steam from other plant operations. Many variations of simple settling have been suggested, but at best they remove but a portion of the water, and are generally supplanted by one of the methods which follow.

(2) *Use of Centrifuges.* This method has been used with more or less success abroad, and only in the case of tars with tars of low free-carbon content, so as to avoid the necessity of frequent cleaning. The tar is first heated to a temperature of 40 to 50° C. and run into the rapidly revolving drum of a centrifugal separator, illustrated in Fig. 106. The tar being heavier than the water is forced to the periphery, the water forming a cylindrical layer inside. An annular diaphragm *A* attached to the upper part of the centrifugal has a ring of perforations where it comes in contact with the drum at *B*. The crude tar is introduced through the pipe *C* below the diaphragm. The speed with which the drum revolves causes the tar to flow through the perforations into the upper portion of the cen-

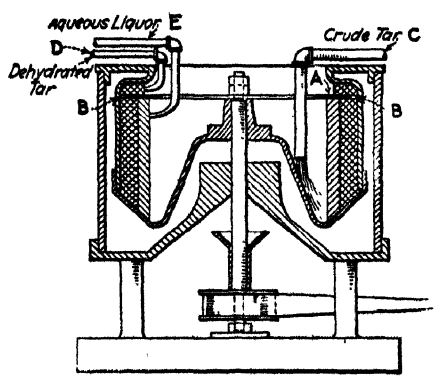


FIG. 106.—Centrifugal Tar Dehydrator.

trifugal, where it is removed through the pipe *D*. The water is drawn off through *E* below the diaphragm, which bars its passage into the upper section. This method is particularly suited for treating tars having approximately the same specific gravity as water, as for example water-gas tar. The centrifugal is revolved at a speed between 2000 and 3000 r.p.m. Tars containing between 30 and 90 per cent of water will have the percentage reduced to less than 1 per cent in one treatment. A large portion of the free carbon contained in the tars is also removed, and affixes itself to the inner walls of the drum from which it must be scraped occasionally.

(3) *Horizontal Stills.* These are used to a considerable extent in Great Britain according to the continuous dehydrating methods proposed by Hird, Chambers and Hammond.[9] They depend upon the use of a horizontal still heated by tubes at the bottom, through which the furnace gases are passed. The tar to be dehydrated is caused to flow through the still to a depth of about 6 in. over the tops of the heating tubes, and a heat-exchanger is used on the incoming wet tar and the outgoing hot dry tar, so as to preheat the former. Two or several stills in series may be employed, depending upon the amount of water carried by the tar and the tonnage to be treated per day.

(4) *Tube Heaters.* This process has been devised by T. O. Wilton,[10] and has met with favor in Great Britain. The tar is treated continuously upon being heated to 170 to 190° C. under a pressure of 30 to 50 lb. per sq. in., by pumping through a coil of pipes in a furnace heated with coke. It is then released into a vapor chamber at atmospheric pressure, whereupon the water and light oils evaporate, since they are maintained at a temperature considerably higher than their boiling points. This is accompanied by copious frothing, due to the fact that each volume of water is converted into 1640 volumes of steam. As this method involves the charging of hot tar to the tube heater, no heat exchanger is used.

(5) *Cascade System.* In Great Britain, this system has been patented by William Blakeley,[11] who allows the heated tar to flow over a series of trays arranged in a casing, the trays being interconnected by pipes which also serve to support them. The tar is introduced in the top tray and is allowed to flow from tray to tray, while the steam or other heating medium is introduced in the pipes at the bottom tray, and is conducted from tray to tray upwards. A development of this process has been used successfully in the United States in a cascade dehydrator, consisting of a rectangular steel chamber enclosing a series of steel pans fitted with steam coils.[12] The pans are set at an incline with alternate pans sloping in opposite directions, so as to form a cascade. Pumps are provided for handling the crude and dehydrated tar, also the distillate. The tar

is introduced through a manifold at the top of the casing which delivers it to a distributing box over the top pan, whence it overflows from pan to pan and leaves the bottom of the casing free from water. The vapors are conducted to a combination vapor-condenser and tar-preheater whence they are condensed by the raw tar on its way to the distributing box. A second water-cooled condenser is provided as a safeguard. The distillate is withdrawn from the second condenser by a vacuum pump, which serves to maintain a vacuum of 3 to 4 in. of mercury on the casing. The dehydrator is operated so that the exit tar attains a temperature of 275 to 285° F., which assures its complete dehydration. A sufficient pressure is applied to the tar before entering the casing to prevent the evolution of vapors, this particular feature being similar to the Wilton system. A dehydrating unit of this type handles 37,000 gal. of gas-works coal tar in twenty-four hours, reducing the water content from 7.7 per cent to 0.1 per cent; likewise 60,000 gal. of tar containing 4 per cent water, and 100,000 gal. containing 1.8 per cent water; in both of the latter instances reducing the water content to 0.1 per cent.

Methods of Distilling Coal Tar.[13] Coal tar is transported from the gas-works or coke-ovens in cylindrical steel tank-cars 7 to 8 ft. in diameter and 28 to 30 ft. long holding about 10,000 gal., provided with a dome on top and heating coils inside for the introduction of steam in cold weather to reduce the fluidity. It is transported by water in tank-vessels constructed similarly to those used for petroleum, holding up to 300,000 gal. At the distilling plant, the tar is generally stored in covered vertical cylindrical steel tanks larger in diameter than height, having a capacity up to 2 million gallons. A certain amount of water separates during storage which is tapped through pet-cocks in the side. Less often the tar is stored in rectangular reinforced concrete tanks built underground.

Tar when heated to a temperature, usually in the neighborhood of 700° F., will crack and decompose, giving off permanent gas with the formation of insoluble matter, known as "free carbon." This reaction increases in velocity as the temperature increases, and varies of course with different tars. The effect of this reaction is a smaller yield of distillate to a given consistency of pitch residue, or in other words, it means that the pitch is produced at the expense of the yield of oils. Since the oil is the more valuable product, the cracking is regarded as undesirable. The amount of cracking is a

function of both temperature and time, increasing directly with either of these factors. The ideal system of tar distillation may be regarded as one which exposes the tar to the lowest temperature for the shortest possible time necessary to obtain the desired product. The practical effect of these factors may be exemplified by some results on a coke-oven tar run to a pitch of 300° fusing-point. A reduction in the time of heating from twelve to one and one-half hours resulted in an increase of 16 per cent in the yield of oils and a corresponding decrease in the amount of pitch. Similarly, where the time was kept constant, a lowering of the final still temperature by 200° F. accomplished approximately the same result. Greater yields of oil may be obtained by decreasing both the time and temperature, although the effects are not necessarily additive.

One method consists in increasing the still heating surface, so as to increase the heat input in a given time, and this leads to the continuous tube-still of one type or another, where the time factor is reduced materially over the batch stills. Another method is to reduce the still temperature by lowering the vapor pressure of the oils through the use of steam, vacuum, or inert gases, as will be described presently.

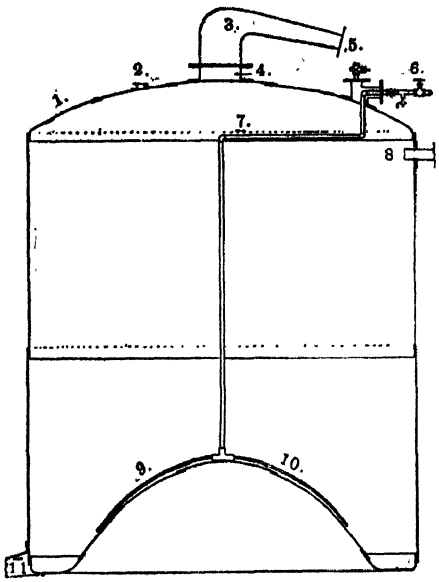
The following methods have been used for distilling coal tars:

(1) *Simple Batch Stills.* The first stills used in the tar industry in Europe were constructed in the form of a "pot" or vertical still having a concave bottom, as illustrated in Fig. 107, where 1 represents the manhole; 2, safety-valve connection; 3, swan neck; 4, swan neck stool; 5, dipping tap; 6, steam inlet; 7, steam pipe; 8, tar inlet; 11, tar outlet. The concave bottom is claimed to have the advantages of providing a larger heating surface, also to assist in draining off the pitch and to accommodate the expansion and contraction of the metal plates without setting up dangerous strains. Steam is introduced during the process of distillation through a perforated pipe (9–10) at the bottom and serves to carry off the vapors more rapidly, also reduce the time of distillation. English stills hold 20 to 40 tons (4000 to 6000 gal.) of tar, and are mounted on suitable brick settings adapted for direct heating with coal or producer gas.

The vapors leave the still through a large pipe connected with the condenser coils immersed in water in a rectangular tank. These coils are constructed of pipes ranging from 6 in. down to 3 in. in diameter. The distillate is run into small measuring tanks which in turn empty into large storage tanks. Each fraction is caught

separately, four fractions all told being recovered, viz.: light oil or crude naphtha, middle or carbolic oil, heavy or creosote oil and anthracene oil.

In the United States the customary form of batch still consists of a horizontal cylinder with convex ends, heated directly with oil, coal or gas, constructed to hold in the neighborhood of 50 tons (10,000 gal.) tar. Such stills are about 9 ft. in diameter by 20 ft. long, and about half the diameter, or 500 sq. ft., is available as heating surface. They take ten to twenty hours to complete distilling a charge to a pitch, where 40 to 45 per cent of oil is removed from the tar. A typical still is illustrated in Fig. 108. The tar enters through a pipe into the top of the still, and the vapors are drawn off through another pipe of large diameter attached directly to the top of the still, at the center. The stills are not usually provided with domes, as is the case with petroleum stills. The outlet pipe for the pitch is located at the bottom, together with inlet pipes for steam or air agitation. The stills are mounted on a brick setting provided with a fire arch to insure uniform heating, and prolong the life of the bottom. The process is an intermittent one. After the tar has been distilled to the desired fusing-point or consistency, the residue of pitch is discharged by gravity, pumping or blowing out with air.



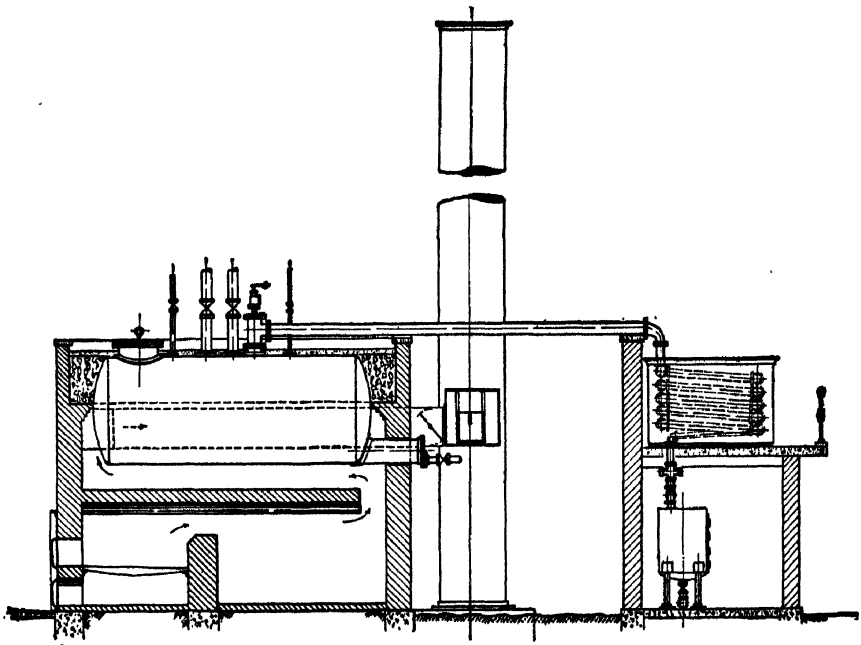
From "Coal Tar Distillation," by A. R. Warnes.

FIG. 107.—Vertical Still for Refining Coal Tar.

In recent years the batch stills have been somewhat improved by the use of internal flues to increase the heating area and serve to reduce the time of distillation, also to minimize the carbon deposition. A modern installation is illustrated in Figs. 109 to 113, consisting of two primary stills of 9000 gal. each and three secondary stills of 8000 gal. each. The primary stills are of the vertical type and serve as preheaters, since they are equipped with spiral steel flues through which the gases from the secondary stills are passed, as illustrated in Fig. 111. From the primary stills the heated tar is run into the secondary stills, where direct heat is applied, and the bulk of the distillation takes place; these are shown in Fig. 112.

The pitch is run from the secondary stills to the pitch coolers of about 5000-gal. capacity each, which are illustrated in Fig. 113. The vapors are condensed in a vertical shell-and-coil type of condenser filled with water, which is located directly below the primary stills, from which the distillate is run into the storage tanks.

Another arrangement consists in operating the batch still in connection with a dehydrator of the continuous type, which has the



Courtesy of The Barrett Company.

FIG. 108.—Horizontal Still for Refining Coal Tar.

advantage of conserving heat and also obviates the danger of foaming during the distillation process.

(2) *Vacuum Distillation.* Vacuum of 26 to 28 in. mercury may be used to advantage in connection with batch stills when running to a hard pitch of 280–300° F. fusing-point. Much better yields of oil are obtained (e.g., about 10 per cent at a 300° F. fusing-point pitch) than by distillation under atmospheric pressure, and the reduction in cracking also has the effect of lowering the percentage of free carbon in the pitch. In general, the use of vacuum does not materially influence the products until a pitch of 140° F. fusing-point is reached, but from there on the vacuum has decided advantages as compared with ordinary operations, although when

the fusing-point of the pitch reaches a fusing-point of 300° F. or thereabouts, a certain amount of cracking begins, accompanied by the evolution of gas, which makes it impractical to maintain the

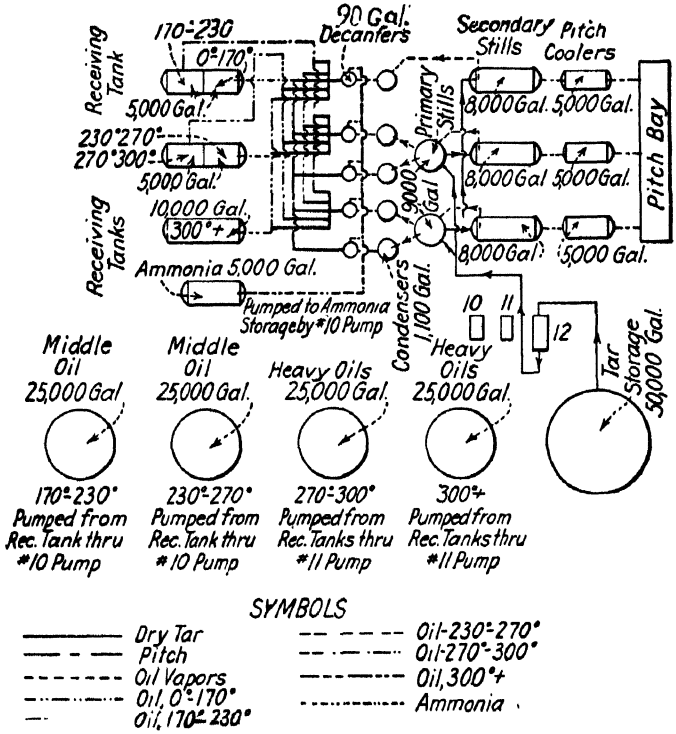


FIG. 109.—Flow Diagram of Dry Tar to Light and Heavy Oils.

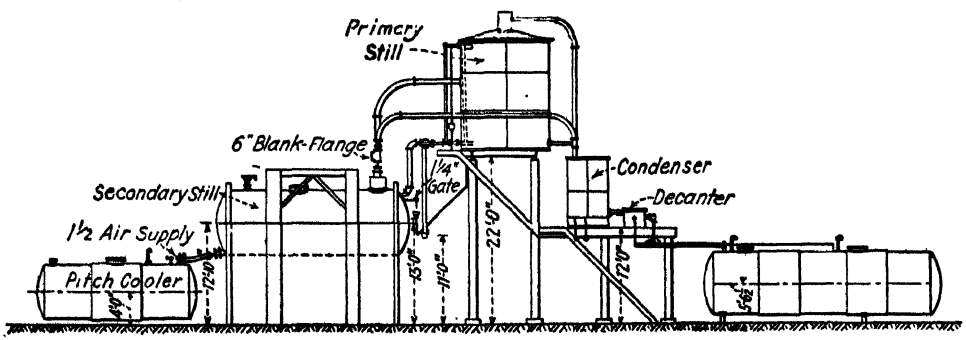


FIG. 110.—Cross-section of Tar Distillation Plant.

necessary degree of vacuum. In distilling to a pitch of 220° F. fusing-point, the final still temperature is 750° F. at atmospheric pressure, in comparison to 570° F. when distilled under a vacuum

of 28 in. mercury. In a vacuum distillation plant, the oil receivers must be in parallel, connected with suitable piping, so that one may be released to empty it, without disturbing the vacuum on the rest of the system.

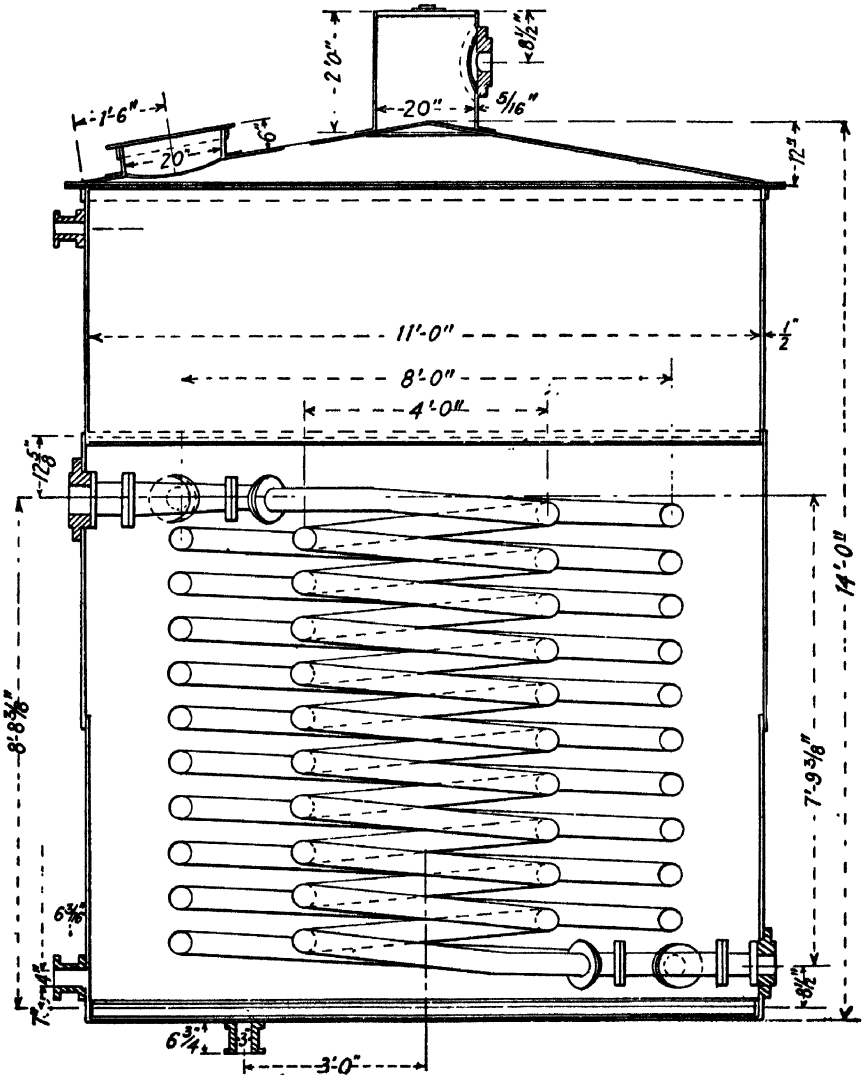


FIG. 111.—Primary Tar Still.

An alternate procedure consists in agitating the tar with steam, using a vacuum of about 15 in. of mercury. The following figures were obtained on coke-oven tars of 9 per cent free carbon:

	Final Still Temp.° C.	Per Cent by Weight		Pitch Tests	
		Pitch	Oil	Air M. Pt.	Free Carbon
Atmospheric.....	400	64.3	34.9	220° F.	35.3 per cent
Vacuum of 28-in....	300	55.9	43.1	216° F.	26.1 per cent

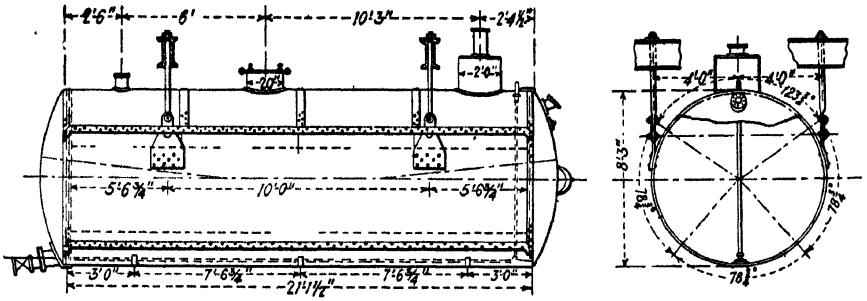


FIG. 112.—Direct-fire Secondary Tar Still.

In general, the softer the pitch produced, the less will be the advantage of vacuum distillation.

Heating in a bath of molten metal under vacuum has also been suggested.[14]

(3) *Steam Distillation.* The passage of superheated steam through the contents of the batch still also serves to reduce the dis-

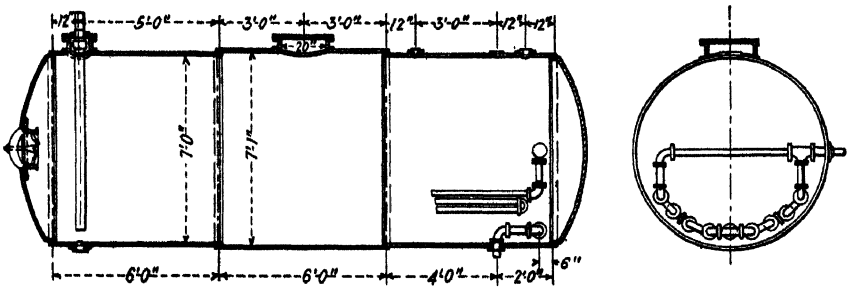


FIG. 113.—Pitch Cooler.

tillation temperature and to minimize the cracking,[15] but the amount of steam required is so great, that its use has not proven practicable from the standpoint of cost and on account of the greatly increased condenser surface required. This, however, does not apply to the use of a small amount of steam to agitate the contents of the still, where the distillation takes place at atmospheric pressure.

The effect of large volumes of steam is indicated by the follow-

ing figures,[16] where steam was passed at a rate of 0.11 cu. ft. (corrected at 77° F. to 760 mm.) per gallon of tar per minute, applicable to a coke-oven tar of 8.5 per cent free carbon content:

	Final Still Temp. ° C.	Per Cent by Weight		Pitch Tests	
		Pitch	Oil	Air M. Pt.	Free Carbon
Ordinary distillation.	413	57.8	40.1	280° F.	40.7 per cent
Steam distillation...	340	47.0	50.6	279° F.	28.8 per cent

It may be noted that the steam consumption was such that the condensed water approximately equaled the volume of the "oil" recovered on distillation.

(4) *Gas Recirculation.* By circulating an inert gas such as nitrogen or carbon dioxide through the contents of the regular batch stills at the rate of 1 to 20 cu. ft. per 100 gal. tar, per minute, the vapor pressure of the oil vapors is lowered, coupled with a reduction of the still temperatures and an increase in the yield of distillate.[17] This procedure merely involves the addition of a gas recirculation pump and closed distillate receivers to the batch-still equipment. In practice, no provision is made for the inert gas, as the system may be filled with air at the start, since in a short time the oxygen is used up and the recirculation gas becomes nitrogen. The formation of free carbon is minimized, and the distillation may be carried to a much further point without danger of coking of the still contents. Whereas pitches of about 300° F. fusing-point represent the hardest that may be produced commercially by the foregoing distillation methods, with the recirculation method, pitches of 400 to 450° F. fusing-point may readily be obtained, and with a correspondingly greater yield of distillate. Thus, with a coke-oven tar of 8.5 per cent free carbon content, 65 per cent by weight of distillate was obtained upon running to a pitch of 400° F. with a carbon deposit in the still no greater than was the case upon running to a 250° F. fusing-point pitch by the ordinary method, with only 40 per cent distillate.

A comparison of the oil yield by the gas-recirculation method on the same type of coke-oven tars referred to under the captions "Vacuum Distillation" and "Steam Distillation," is given below. The gases were recirculated at a rate of 0.33 cu. ft. (corrected to 77° F. and 760 mm.) per gallon of tar per minute, in the case of CO_2, and at the rate of 0.40 cu. ft. (corrected to 77° F. and 760 mm.) per gallon of tar per minute, in the case of N_2:[16]

	Final Still Temp. ° C.	Per Cent by Weight		Pitch Tests	
		Pitch	Oil	Air M. Pt.	Free Carbon
Atmospheric.....	413	57.8	40.1	280° F.	40.7 per cent
Recirculated CO_2....	358	46.9	52.6	277° F.	29.0 per cent
Recirculated N_2.....	331	46.4	52.0	277° F.	27.2 per cent

(5) *Continuous Stills.* Various expedients have been proposed [18] to transform the batch distillation process into a continuous operation, as for example placing several stills in series and flowing the tar through them consecutively and taking different fractions from each still, allowing the pitch to flow from the last in the series. In Great Britain two continuous systems of tar distillation are in use, as devised by H. P. Hird and E. V. Chambers respectively. The Hird process [19] involves the use of four stills, three of which are connected in series, the first two being heated by internal flues and the third being arranged for the introduction of steam. The fourth still is used for the production of pitch. Separate condensers for each still and suitable heat exchangers are provided. The first still is maintained at 190° C., the second at 240° C., the third at 325° C. (producing creosote oil), and the fourth at 275° C. The stills are provided with a series of vertical baffles and the tar flows through the system in a comparatively thin layer. The Hird system is only adapted to producing pitches ranging in fusing-point up to 200° F.

The Chambers process is a modification of the cascade system used for dehydrating tars. It involves the use of two stills provided with cascades, three condensers and one heat interchanger. The crude tar is preheated by flowing through the heat interchanger, and then caused to flow down the cascade in still No. 1, followed by a return through the bottom of the still which is kept about one-third full of tar. The tar then passes through the still No. 2 in a similar manner, and finally into Still No. 3 which is not equipped with a cascade, where the residue is distilled to pitch with the introduction of steam. Still No. 1 is maintained at 210° C. and produces light oil, still No. 2 is kept at 260° C. and produces light oil and creosote, still No. 3 is kept at 320° C. and produces anthracene oil and a residue of pitch. It is claimed that a plant of this type will successfully handle a Mond or water-gas tar containing as much as 30 per cent of water.

(6) *Tube Stills.* These operate on the principle of heating the tar by passing it through a coil of pipe surrounded by the heating medium, and then allowing the vaporization of the oils to take

place in a vapor chamber at the exit of the coil, thus handling the tar continuously.

In Great Britain the Wilton process [20] has met with much favor, and consists of a modification of the method described previously for dehydrating tar. The Wilton tube-still for the continuous distillation process consists of two successive heating coils in series, followed by a cylindrical still in which superheated steam is introduced, with expansion chambers in between each unit. The first coil heats the tar to 200° C. under a pressure of 30 to 50 lb. per sq. in. and removes the light oils and water. The second coil heats the tar to 230–300° C. and removes the creosote oil. The last still removes the anthracene oil by means of the superheated steam at 300° C., and separates the pitch.

Other types used in Europe include the so-called "Sadenwasser" [21] and "Leinweber" [22] stills, named after the makers.

Similar stills are in use in the United States with very satisfactory results.[23] They utilize a single heating coil when running to pitches of about 200° F. fusing-point, and two heating coils when running to a 300° F. pitch, which is about as hard as can be produced in this type of apparatus. The crude tar is first introduced as cooling means in the various oil condensers, whereby it is heated sufficiently, so that when released into an expansion chamber, the water and light oils are vaporized and condensed, whereupon the dehydrated tar is pumped through the first heating coil. After passing through the first coil, the tar is sprayed into a second expansion chamber where the creosote oil is taken off to fractional condensing means, and the residual intermediate pitch pumped through a second coil in the same furnace as the first coil, but in a hotter zone, and the same process repeated. For the softer grades of pitch the second stage may be omitted. The use of vacuum in the second stage will serve to still further reduce the operating temperatures.[24] The smallest still of this type handles from 30,000 to 50,000 gal. of tar per day, depending upon the character of the tar and the grade of pitch desired, whereas the largest unit in operation in the United States has a capacity of 90,000 to 140,000 gal. tar per day. The yield of oils in such stills will run 10 to 15 per cent better than the ordinary batch still in producing pitch of 300° F. softening-point, due entirely to a reduction in the time of heating to less than one-fifteenth that required in the latter. The general flow relationship of a well-developed tube-still installation in the United States is shown in Fig. 114.

Continuous fractional condensing columns have been developed which separate the vapors into sharper fractions than were previously obtainable in the batch stills. Furthermore, a number of closely cut fractions may be obtained simultaneously and continu-

ously from the mixed tar vapors, in a properly designed and constructed condensing column, including the separation of creosote oil, naphthalene oil, heavy oil and anthracene oil.

(7) *Collector-main Condensers.* A process has been developed [25] in the United States, in which tars of the desired consistency may be separated directly in the collector-main of the coke-oven, by scrubbing the hot vapors with the preheated tar, or else with some of the recovered oils, which are introduced in the form of a spray. These serve to remove the tarry matter from the vapors, which are

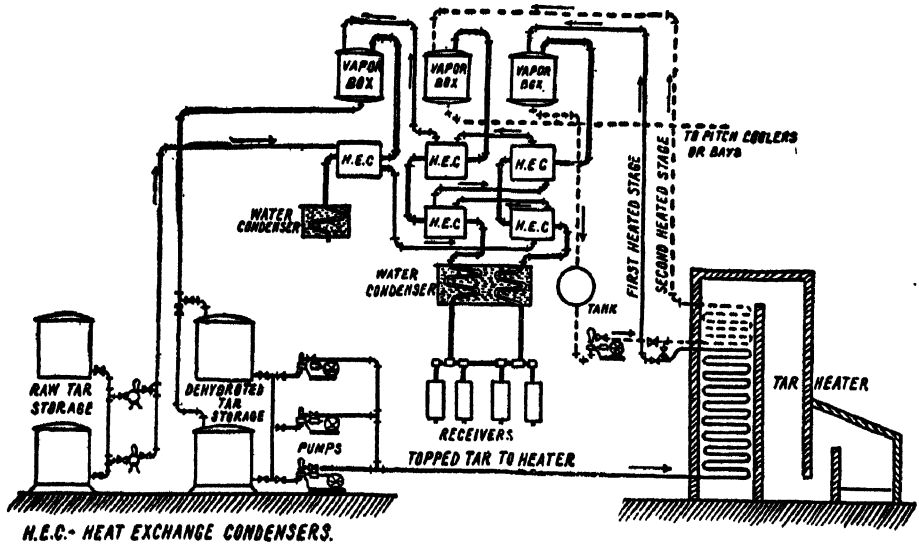


FIG. 114.—Flow Diagram of Continuous Tube-still for Coal Tar.

thereby cooled and at the same time the gases are enriched. The equipment is illustrated diagrammatically in Fig. 115.

The process operates as follows: the hot gases from the ovens pass up the ascension pipes, 1, into the collector main, 2, which is connected at one end with the foul gas or air-cooled main 3. The two mains have a continuous fall in the direction of gas flow, and tar is circulated through them by the pump, 4, and gravitates back to the tank, 5, accompanied by the heavier tar fractions which have condensed from the gas.

The essentially valuable feature of the process is that road tar of any required standard can be withdrawn from the air-cooled or foul gas main at point 6, at which the temperature of the gas is still above the dew point of the lighter tar constituents, which are carried forward with the gas. By a simple manipulation of this fundamental physical condition of the foul gas stream, road tar

may be produced, continuously or intermittently, the tar so obtained being superior in essential respects to that produced by dehydration or distillation in stills. Perfectly dry road tar may be run while hot from the seal pot, 12, to the receiver, 11, where the viscosity is checked and adjusted, if necessary, by mixing with oil or road tar of suitable consistency.

With a pitch run to 300° F. fusing-point, a 70 per cent oil yield is claimed, as compared with 44 per cent in a batch still. Creosote oil as well as hard pitch may be collected in a similar manner. Hard pitch may be burnt as fuel, or returned to the coke-ovens with the charge of coal.

This process represents quite a forward step, and is rapidly being adopted in by-product coke-ovens, both in the United States

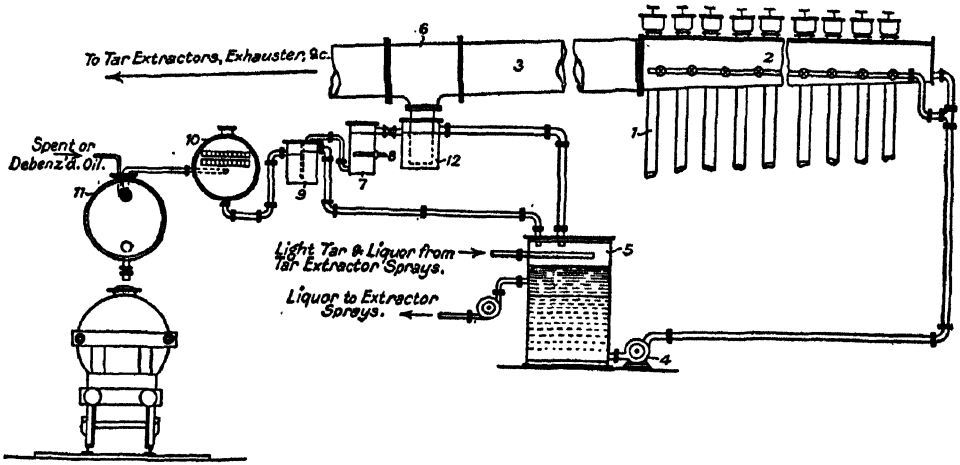


FIG. 115.—Collector-main Condenser for Coal-tar Distillation.

and abroad, since it is both more efficient and economical than the use of condensers and scrubbers.

Recovery and Treatment of Coal-tar Distillates. On refining coal tar by any of the distillation processes previously outlined, one or more of the following distillates are obtained:

(1) *Light Oil.* At the commencement of the distillation process, the light oil or "crude naphtha" distils over, and comprises the entire distillate lighter than water, being obtained below a vapor temperature of 200° C. This fraction is first washed with alkali to remove the tar acids, and then redistilled and fractioned into crude benzol, toluol, solvent naphtha (xylol) and heavy naphtha.

(2) *Middle Oil.* This is generally considered to include the fraction boiling between 200 and 250° C. In some cases it is mixed

with the ensuing fraction—the heavy oil—provided the mixture will meet existing specifications for creosote oil. In other cases the middle oil is first extracted with caustic soda to remove the tar acids before it is mixed with the heavy oil, for use as creosote. Still another alternative consists in redistilling the middle oil in fire-heated stills to separate it into the following fractions: crude naphtha up to 210° C., acid-naphthalene oil from 210 to 250° C., and creosote oil above 250° C. The crude naphtha is treated with caustic soda to remove the tar acids and then worked up with the light oil fractions. The acid-naphthalene oil is first cooled in pans to crystallize out the naphthalene, whereupon it may either be marketed as such under the designation "tar-acid oil," according to the percentage of tar acid content (e.g., 15 per cent, 25 per cent, 50 per cent, etc., as the case may be), or it may have the tar acids removed and marketed as naphthalene oil.

In Germany, the practice consists in distilling the middle oil in a fire-heated vacuum column still, which gives much closer boiling fractions, as follows: crude heavy naphtha up to 200° C., a second fraction from 200 to 220° C. containing the bulk of the cresylic acid and comparatively little naphthalene, next a closely cut naphthalene oil fraction carrying a high percentage of crude naphthalene, leaving a residue of so-called creosote oil.

The tar acids extracted from the various fractions obtained principally from the middle oils are obtained as their sodium salts, which in turn are treated with carbon dioxide to liberate the tar acids, phenols, etc. These acid products are separated and marketed under the following designations: phenol, cresols, 97–99 per cent straw-colored cresylic acid, 95 per cent dark cresylic acid, ortho-cresol, meta-cresol, para-cresol, etc. They are variously used for the production of emulsifiable disinfectants, cattle sprays, and as flotation oils for the concentration of certain grades of mineral oils.

(3) *Heavy Oil.* This is variously known under the terms heavy oil, creosote oil and dead oil, and may either represent the fraction between 250° C. to the end of the distillation, or the fraction between 250° C. and the anthracene oil fraction. United States practice consists primarily in adjusting the heavy oil fraction to meet the various specifications established for creosote oil for use in wood preservation.[26] This may be accomplished by controlling the distillation range of the fraction, or by mixing together various fractions obtained from the middle and heavy oil respectively. In either case, it is advisable to remove any naphthalene by cooling and filtering.

(4) *Anthracene Oil.* Unless it is intended to extract anthracene, the anthracene oil fraction is not made, and the entire cut is

worked up for creosote oil. Where it is intended to produce anthracene, the anthracene oil fraction is cooled in pans or tanks until the anthracene crystallizes out, whereupon it is filter-pressed to obtain a cake containing 20–30 per cent anthracene, or else subjected to a hot hydraulic pressing to remove more oil and produce a cake containing 30–40 per cent anthracene. The anthracene is used principally to produce alizarin and other coal-tar dyes.

Recovery and Treatment of Coal-tar Residuals. At the close of the distillation process the residual product consists either of coal-tar pitch, or else refined coal tar, depending upon its physical characteristics. There is no sharp line of demarcation between the two, although, in general, residuals having a fusing-point above 80° F. (cube method—Test 15*c*) are known as pitches, whereas those fusing below are considered refined coal tar. The characteristics of a given residual will vary with the nature of the tar, the particular method of distillation, and the quantity of distillate removed. When the distillation has been completed, the hot pitch is run into coolers illustrated in Fig. 113, and when the temperature falls to 250 to 300° F. the product is either run into barrels or into a large rectangular concrete enclosure known as a "pitch bay," where it is allowed to solidify. Various methods have been suggested to facilitate the handling of hard pitches,[27] consisting in molding it,[28] rapidly cooling the pitch and breaking it into fragments,[29] comminuting the product,[30] dropping the melted pitch from an elevation into a body of water,[31] forming an aqueous suspension with a peptizing agent,[32] etc.

If the distillation is continued to the desired point, then the residue is known as "straight-run coal-tar pitch." On the other hand, if the distillation is carried to a point where the residue is harder and more infusible than desired, and is thereupon fluxed to the desired consistency and fusing-point, either with certain fractions of the distillate [33] or a flux of different origin—usually of little value commercially—or with other tars,[34] then the pitch is known as "cut-back coal-tar pitch." [35]

It has been claimed that coal-tar pitch treated with petroleum distillate (e.g., naphtha) and steam in a heated vessel with agitation, and then being allowed to settle, will separate into (a) a clear supernatant liquid suitable for use as a paint vehicle, and (b)

a pitch-like mass suitable for the manufacture of roofing compounds.[36] Processes have been described in which low-temperature coal tar is distilled to a pitch in the presence of NaOH;[37] likewise the steam-distillation of coal-tar pitch with Fe_2O_3 and $FeCl_3$ in combination with montan wax, whereupon distillates are said to be obtained composed of waxy and resinous products.[38]

The crystallization of naphthalene, anthracene, phenanthrene, etc., from coal tars and their distillates may be inhibited by adding a small percentage of stearyl chloride.[39]

Coal-tar residuals have found many uses in the arts, among which may be cited the following: very soft pitches and refined coal tars are used for saturating felts; for dust-laying purposes on roads and pavements; as the base of various kinds of paints and protective coatings for metals, masonry, and other structural materials. Moderately soft pitches having a fusing-point between 80 and 120° F. are used principally for road binders and sometimes for waterproofing work. Medium hard pitches having a fusing-point between 120 and 160° F. are used for constructing "pitch-and-felt roofs"; for laminated membranes used in waterproofing foundations of buildings, tunnels, subways and bridges; as pipe-dips; as a binder in constructing bituminous concrete and macadam pavements; for filling the joints in block pavements; and for manufacturing the better grades of bituminous paints. Hard pitches having a fusing-point between 160 and 212° F. are used principally as a binder for fuel briquettes. Very hard pitches having a fusing-point above 212° F. are employed as a binder for sand-cores in forming castings of iron and steel; for manufacturing electric-light carbons, battery carbons, carbon brushes for motors and dynamos, black "clay pigeons" for target shooting, and sundry plastic compositions for insulating purposes, as a filler for rubber goods,[40] etc. It has been pointed out[41] that very hard coal-tar pitches (fusing-point between 300 and 400° F.) may be cut-back upon melting with pitches of a lower fusing-point.

Coal-tar pitches are remarkably resistant to the disintegrative action of water, and are therefore well adapted for sub-soil waterproofing. They are more weather resistant than wood-tar pitch, rosin pitch, lignite-tar pitch, shale-tar pitch and bone-tar pitch, but

are inferior to carefully prepared residual asphalts obtained from petroleum, blown petroleum asphalts, wurtzilite asphalt, fatty-acid pitches and pure native asphalts containing approximately the same percentage of volatile matter.

The manufacture of pitch-coke has assumed considerable importance in recent years, for use in the metallurgical industries, especially in the production of certain high-grade castings, on account of its extreme purity and freedom of deleterious mineral constituents which are present in coke produced directly from coal, also for manufacturing electrodes. Pitch-coke results when coal-tar pitch is distilled in a retort until all the volatile ingredients have been driven off. As the distillation proceeds beyond the anthracene oil, the character of the distillate assumes a grease-like consistency, which thickens as the distillation proceeds. At the same time a strong evolution of gas (mainly hydrogen) takes place, accompanied with a certain amount of ammoniacal liquor. The residue at this stage is very pasty and of a high fusing-point and there is considerable danger of the still foaming over. As the heating is continued, the mass cokes and the distillate appears as a transparent ruby-red resinous substance, having a specific gravity above 1.22, known as "pitch resin," which melts between 40 and 100° C.[42] The temperature at the close of the distillation ranges between 800 and 1000° F. The yield of resin may be increased by distilling the pitch with steam in the presence of Fe_2O_3 and $FeCl_3$.[43] Another type of pitch resin (fusing-point 105–110° C.) may be obtained from the sludge produced in refining the light oils with sulfuric acid, which is neutralized with caustic soda and distilled to 225–300° C. under a vacuum of 28 in.[44] The character of the pitch-coke varies, depending upon the type of apparatus in which the pitch is coked, the time of heating, and the final temperature attained. It consists of practically pure carbon, except for 0.2 to 0.5 per cent ash. In Europe the distillation is performed in special cast-iron pot stills with large doors or man-holes to facilitate cleaning, holding 1 to 2½ tons of pitch, which are run on a thirty-six hour cycle. The resulting coke is quenched with water and then chopped out of the still by hand. The pitch charged usually has a fusing-point of 60 to 75° C., and the products ob-

tained include 30 to 40 per cent of an orange-colored waxy grease (specific gravity 1.14 to 1.22); 4 to 6 per cent of pitch-resin; traces of ammonia; and 50 to 60 per cent of pitch-coke.

In the United States a large quantity of pitch-coke has been produced by first preparing a pitch having a fusing-point of 400 to 450° F. by the recirculation method of distillation,[45] which is then coked in a bee-hive type of coking oven,[46] no attempt being made to recover the by-products which are employed to furnish the heat required for the coking operation. The pitch used has a fusing-point (cube method—Test 15*c*) of 440° F. and fixed carbon (Test 19) 63 per cent. A charge of 6.5 tons may be coked in sixty-four hours at a maximum temperature of 1100° F. The coke is then quenched with water and removed, whereupon it contains 1 per cent of volatile constituents, 0.48 per cent ash and 0.38 per cent sulfur, being approximately 98 per cent pure carbon. The usual unit is a 5000 gal. still with a 4000 to 4500 gal. charge, operating on a 48 hour cycle, yielding 60 to 75 per cent oil from coke-oven tars. A modified type of pot-still is also used, consisting of a cylindrical still set horizontally in brickwork, so that its entire circumference is surrounded by the hot gases, thereby preventing condensation and speeding the distillation process.[47] Alternate procedures consist in distilling in vacuo; mixing the pitch with coke breeze and charging same into the coke ovens;[48] spraying the melted pitch with steam into the coke-oven chamber;[49] etc. A cellular pitch coke suitable for use as a decolorizing agent may be obtained by distilling coal-tar pitch with NaOH, KOH, MgO, or CaO.[50]

The successful coking of pitch has provided the necessary outlet for the surplus production, which cannot be disposed of through the regular channels.

Table XXIV gives a schematic outline of the various products derived from coal upon distilling it destructively, including the commercial substances produced from coal tar, as practiced in the United States.

Properties of Coal-tar Pitches. The figures in Table XXV will serve to give a general idea of the limiting physical characteristics of the principal types of coal-tar pitch:

Church and Weiss examined representative specimens of coal-tar pitches,[51] with the following results, in which *A* represents gas-

TABLE XXIV

PRODUCTS DERIVED FROM COAL

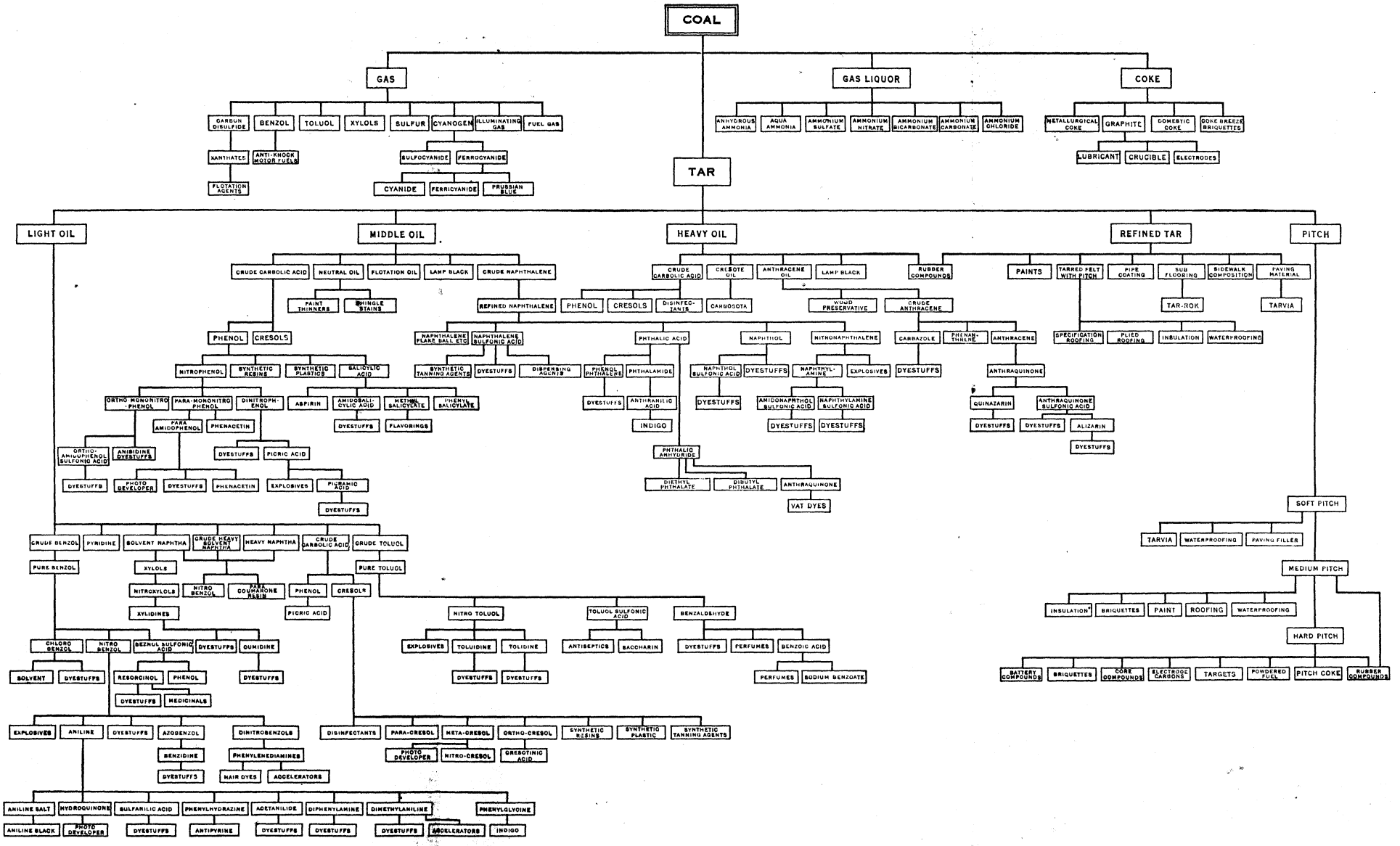


TABLE XXV

	Gas-works Coal-tar Pitches			Coke-oven Coal-tar Pitch	Blast Furnace Coal-tar Pitch	Gas-producer Coal-tar Pitch	Low-temperature Coal-tar Pitch
	Horizontal Retorts	Inclined Retorts	Vertical Retorts				
(Test 1) Color in mass	Black	Black	Black	Black	Black	Black	Black
(Test 2a) Homogeneity to the eye at 77° F	Variable	Variable	Variable	Variable	Variable	Variable	Variable
(Test 2b) Appearance under microscope	Lumpy	Lumpy	Sl. lumpy	Sl. lumpy	Lumpy	Lumpy	Sl. lumpy
(Test 3) Appearance surface on aging	Unchanged	Unchanged	Unchanged	Unchanged	Dull	Unchanged	Dull
(Test 4) Fracture	Conchoidal	Conchoidal	Conchoidal	Conchoidal	Conchoidal	Conchoidal	Conchoidal
(Test 5) Lustre	Variable	Variable	Variable	Variable	Variable	Variable	Variable
(Test 6) Streak on porcelain	Black	Black	Black	Black	Black	Black	Black
(Test 7) Specific gravity at 77° F	1.25–1.40	1.25–1.35	1.15–1.30	1.20–1.35	1.20–1.30	1.20–1.35	1.10–1.26
(Test 9b) Hardness at 77° F. (needle penetrometer)	0–100	0–100	0–100	0–100	0–100	0–100	0–100
(Test 9d) Susceptibility index	> 100	> 100	> 100	> 100	> 100	> 100	> 100
(Test 10) Ductility at 77° F. (cm.)	Variable	Variable	Variable	Variable	Variable	Variable	Variable
(Test 15c) Fusing-point (cube method)° F	80–212*	80–212*	80–300*	80–300*	80–212*	80–212*	80–200*
(Test 16) Volatile at 325° F., 5 hrs. (per cent)	1–10	1.10	1–10	1–10	1–10	1–10	1–10
Volatile at 500° F., 5 hrs. (per cent)	3–20	3–20	3–20	3–20	3–20	3–20	3–20
(Test 17b) Flash-point (open tester), ° F	250–450	250–450	250–450	250–450	250–450	250–450	250–450
(Test 19) Fixed carbon (per cent)	35–65	30–45	15–40	17–60	10–30	25–45	8–22
(Test 21) Soluble in carbon disulfide (per cent)	45–70	63–78	70–94	50–92	45–75	60–85	85–98
Non-mineral matter insoluble (per cent)	30–55	28–37	6–30	8–50	15–35	15–40	2–15
Mineral matter (per cent)	0–½	0–½	0–½	0–½	10–20	0–2	0–3
(Test 22) Carbenes (per cent)	2–10	2–10	2–10	2–10	1–5	2–10	1–5
(Test 23) Soluble in 88° petroleum naphtha (per cent)	10–20	15–30	20–40	10–30	5–25	10–30	25–60
(Test 24) Free carbon (per cent)	30–55	25–40	5–35	6–40	15–35	15–40	2–25
(Test 28) Sulfur (per cent)	½–1	½–1	½–1	½–1	½–1	½–1	½–1
(Test 31) Tar acids (distillate to coke) (per cent)	5–20	10–15	20–30	1–12	20–30	10–15	25–50
(Test 32) Naphthalene (per cent)	Tr.–2	Tr.–2	0–Tr.	1–2½	0–Tr.	0–Tr.	0–Tr.
(Test 33) Solid paraffins (per cent)	0	0	0–Tr.	0	2–5	0–Tr.	2–5
(Test 34a) Soluble in conc. H_2SO_4 (per cent)	95–100	90–95	90–95	95–100	85–100	90–95	85–90
(Test 34b) Sulfonation residue of distillate 235–315° C. (per cent)	0–3	2–6	4–6	0–5	15–25	0–5	5–20
(Test 37c) Saponifiable matter (per cent)	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1	Tr.–1
(Test 39) Diazo reaction	Yes	Yes	Yes	Yes	Yes	Yes	Yes
(Test 40) Anthraquinone reaction	Yes	Yes	Yes	Yes	No	Yes	No

* Figures given above apply to commercial grades of pitch. It should be noted, however, that coal-tar pitches can be produced with fusing-points as high as 450° F.

works coal-tar pitch obtained from horizontal retorts, *B* gas-works coal-tar pitch from inclined retorts, *C* gas-works coal-tar pitch from vertical retorts, *D* Otto-Hoffman coke-oven coal-tar pitch, *E* Semet-Solvay coke-oven coal-tar pitch, and *F* Scotch blast-furnace coal-tar pitch.

TABLE XXVI

		A	B	C	D	E	F
(Test 7)	Sp. gr. at 60°/60° F.	1.30	1.28	1.19	1.25	1.25	1.23
(Test 9*b*)	Hardness at 115° F.	Too soft	Too soft	Too soft	Too soft	Too soft	324
	Hardness at 77° F.	39	40	44	41	39	41
	Hardness at 32° F.	2	2	2	3	2	8
(Test 15*c*)	Fusing-point (cube method)° F.	125	123	125	126	126	135
(Test 19)	Fixed carbon (per cent)	41.5	37.0	16.3	28.5	28.2	14.4
(Test 21)	Sol. in carbon disulfide (per cent).	64.9	67.8	89.5	79.9	82.5	58.2
	Non-mineral matter insoluble (per cent).	34.9	32.0	10.3	19.7	17.4	30.0
	Mineral matter (per cent).	0.2	0.2	0.2	0.4	0.1	11.8
(Test 22)	Carbenes (per cent).	3.5	2.8	5.3	7.4	5.9	2.6
(Test 31)	Free carbon (per cent)	34.9	30.8	8.0	21.1	18.3	28.4

A sample representing a well-advertised brand of straight-run gas-works coal-tar pitch marketed for built-up roofs was tested by the author with the following results:

(Test 7)	Specific gravity at 77° F.	1.25
(Test 9*b*)	Hardness at 115° F.	95
	Hardness at 77° F.	20
	Hardness at 32° F.	0
(Test 9*c*)	Consistency at 115° F.	5.0
	Consistency at 77° F.	24.7
	Consistency at 32° F.	> 150
(Test 9*d*)	Susceptibility index.	> 100
(Test 10*b*)	Ductility at 115° F.	35
	Ductility at 77° F.	75.5
(Test 9*d*)	Ductility at 32° F.	0
(Test 11)	Tensile strength at 115° F.	0.15
	Tensile strength at 77° F.	4.65
	Tensile strength at 32° F.	8.5
(Test 15*c*)	Fusing-point (cube method).	122° F.
(Test 16)	Volatile matter, 500° F. in 5 hrs.	8.2 per cent
(Test 17*b*)	Flash-point.	360° F.

The consistency, tensile strength (multiplied by 10) and ductility curves of this specimen are shown in Fig. 116.

Coal-tar pitches are characterized as follows:[52]

(1) Jet-black streak on porcelain.
(2) Carbonaceous matter clearly visible under microscope.
(3) Comparatively high specific gravity.
(4) High susceptibility index. This means that they are largely influenced by changes in temperature, becoming brittle in winter, and softening under extreme summer heat.

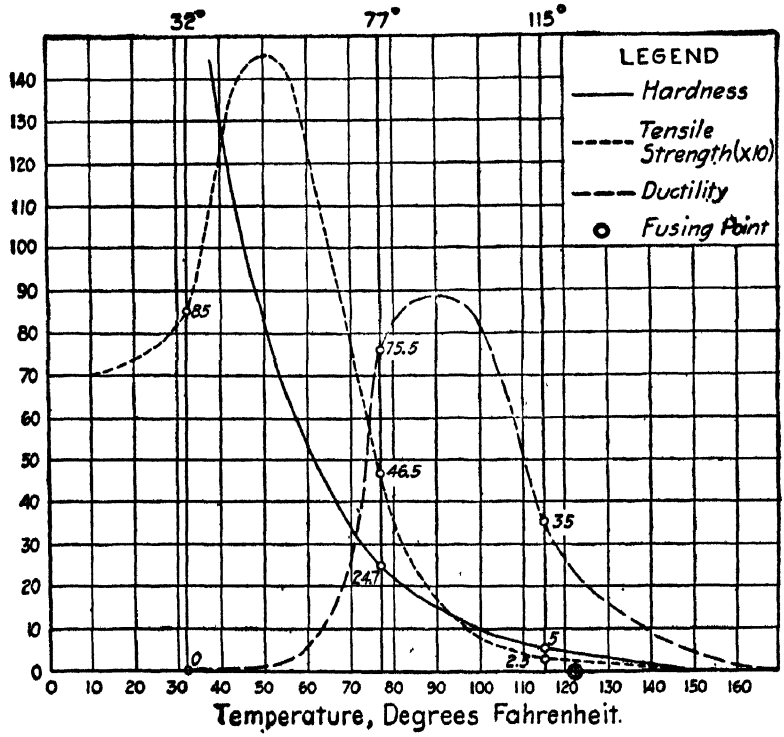


FIG. 116.—Chart of Physical Characteristics of Coal-tar Pitch.

(5) High ductility when tested at temperatures ranging between the solid and fluid states.
(6) Characteristic odor on heating.
(7) Pass rapidly from the solid to the fluid state.
(8) Comparatively high percentage of volatile constituents when heated at 500° F. for five hours.
(9) Comparatively high percentage of fixed carbon.
(10) Comparatively high percentage of non-mineral matter insoluble in carbon disulfide ("free carbon").
(11) Comparative insolubility in petroleum naphtha.
(12) Comparatively small percentage of sulfur.

(13) Naphthalene present in most instances.
(14) Solid paraffins are either absent or present in small amounts.
(15) The sulfonated constituents are soluble in water.
(16) The distillate to coke contains a comparatively large amount of tar acids.
(17) Give the diazo reaction.
(18) Very slightly soluble in ethyl alcohol, forming a yellowish-brown solution, with an intense greenish-blue fluorescence.

Low-temperature coal-tar pitches may be differentiated from high-temperature pitches, since the former:

(*a*) contain more phenolic bodies.
(*b*) contain solid paraffins.
(*c*) contain traces to no naphthalene and anthracene.

Attempts have been made to oxidize coal-tar pitches in the following ways: heating in air with MnO_2 or HNO_3;[53] heating in air with MnO_2 and NH_4Cl;[54] blowing at an elevated temperature (e. g. 370° C.) by the same process used for treating petroleum asphalts;[55] blowing with air under pressure;[56] blowing with air in the presence of sulfur;[57] blowing with air in the presence of sulfur, CaO and $KClO_3$;[58] blowing with air in the presence of MeCHO and NH_3;[59] blowing with air in the presence of H_2SO_4;[60] blowing with air in the presence of H_2SO_4 and a persulfate or perborate;[61] blowing with air in the presence of Fe_2O_3;[62] blowing with air in the presence of $FeCl_3$ or Fe_2O_3 containing sulfur;[63] treating with oxygen at an elevated temperature in the presence of boric, phosphoric or hydro-iodic acid;[64] blowing with oxygen in the presence of nitrous oxides;[65] first emulsifying a mixture of coal-tar pitch and heavy tar oils with an aqueous solution of Na_2CO_3 and Na_2SiO_3 and then adding BaO_2.[66] Processes have also been described for blowing a mixture of coal-tar pitch with saponified vegetable oil,[67] or with asphalt or lignite-tar pitch.[68]

The presence of comparatively large amounts of volatile constituents results in disproportionately high losses in the blowing operation. Samples tested by the author showed a slight lowering of specific gravity, a decrease in the hardness for a given fusing-point, a decrease in the susceptibility factor (i. e., the material was less affected by changes in temperature) and no appreciable change

in the ductility. Blown coal-tar pitches have not been marketed, nevertheless they warrant further development.

Coal-tar pitches may be vulcanized by heating with sulfur;[69] or a mixture of sulfur and alum;[70] or a mixture of sulfur and $CaOCl_2$;[71] or with an alkaline polysulfide or antimony sulfide;[72] or with spent Fe_2O_3 (obtained in the purification of coal gas) in the presence of $FeCl_3$ or $MnSO_4$;[73] or with S_2Cl_2 (with or without sulfur)[74] or by heating with H_2SO_4;[75] or by heating with H_2SO_4 and subsequently heating to a higher temperature until the acid is decomposed;[76] or with organic nitro- or sulfochloride derivatives;[77] or with S_2Cl_2 or thionylchloride and then combined with phenol-formaldehyde resin;[78] or by treating a mixture of coal-tar pitch and vegetable oil with nitric acid and vulcanizing with sulfur;[79] or by heating a mixture of coal tar and vegetable oils or fats with H_2SO_4.[80] These processes serve to raise the fusing point of the pitch and make the product more resistant to temperature changes. In Germany, sulfurized coal-tar pitch has been marketed under the name "Holzzement" for cementing together layers of coal-tar saturated felt in constructing built-up roofs.[81]

A type of coal-tar pitch suitable for use in constructing steep built-up roofs and which will not run or flow at high sun temperatures when exposed at angles in excess of 30° may be prepared as follows: distilled coal-tar pitch or coke-oven-tar pitch is heated to 550° F. and 2 to 3 per cent of ammonium sulfate or zinc sulfate is incorporated, the heating being continued until dissolved. During this treatment the pitch is hardened and its fusing-point (Test 15*c*: cube-in-air method) raised to about 400° F. It is thereupon cut back with 30 to 40 per cent anthracene oil. The final product, known as "rubber pitch" has a low susceptibility, and tests as follows:

(Test 7)	Specific gravity at 77° F.	1.240
(Test 9*b*)	Penetration at 115° F.	55 to 65
	Penetration at 77° F.	20 to 22
	Penetration at 32° F.	5 to 9
(Test 15*c*)	Fusing-point (cube method)	180 to 190° F.
(Test 31)	Free carbon	33 to 36 per cent.

Coal tars and coal-tar pitches may be deodorized by treating with formaldehyde or paraformaldehyde in the presence of acids or alkalies followed by blowing steam through the heated mass.[82] Coal

tars may be hardened and toughened, and thereby rendered less susceptible to temperature changes, by converting the phenols, cresols, etc., in situ, into formaldehyde condensation products (i.e., phenolic resins) as follows: coal tar (1000 parts) is mixed with formaldehyde (10 parts) and ammonia of sp. gr. 0.880 (5 parts), then digested 8 hours at 70° C. in a closed vessel, whereupon a catalyst (e.g., $CuSO_4$ or $FeSO_4$) is added and the mass blown with air for 10 to 30 hours, during which process the temperature is gradually increased to 193° C. The resultant product has been marketed under the name "Bitural".[83] Similarly, synthetic resins may be produced from the fraction of low-temperature coal tar, distilling between 170 and 230° C. (containing phenols), by heating to 100° C. with aqueous 40 per cent formaldehyde and a basic catalyst, such as caustic soda, pyridine, trimethylamine, or tri-ethylamine, until three layers are formed. The upper layer consists of neutral oils and unaltered phenols, the middle layer is aqueous, whereas the lower layer consists of phenolic resins.[84]

Coal-tar pitch may be treated with chlorine in the presence of a chlorine carrier, which serves to increase its lustre, hardness and fusing-point, resulting in a product which resembles asphalt.[85] An asphalt-like product may also be obtained by mixing coal-tar pitch either with the insoluble constituents precipitated from coal tar by treatment with benzol, or with cumarone resin which has been heated to 270° C.[86]

A process of cracking at 510-750° C. in a tube-still serves to convert coal tars into a product which sulfonates completely upon digesting with H_2SO_4.[87]

The percentage of free carbon in coal tars and coal-tar pitches may be increased by heating under pressure between 700 and 820° F.[88]

The following pitches are produced in small amounts, principally in Germany, as by-products in the refining of coal-tar derivatives:

Anthracene Pitch. (*Anthracene-oil-tar Pitch*). This is obtained as a residue upon the purification of anthracene by distillation, and is characterized by being hard, black and glossy. Anthracene pitch may be hardened by heating with sulfur,[89] or with spent iron oxide obtained in the purification of coal gas.[90] Anthracene

pitch may be used for various purposes, including briquette binders.[91]

Naphthol Pitch. This is obtained in the refining of betanaphthol or naphthylamine.[92] It is a glossy black solid, fusing at about 120° C., and is almost completely soluble in solvent naphtha (xylol) chloroform and pyridine. Upon boiling with aqueous caustic soda it yields naphthol, which may be identified qualitatively. Its fusing-point may be increased by heating with formaldehyde in the presence of a mineral acid,[93] and a rubber-like product is obtained upon fluxing with a vegetable oil and adding fillers.[94] Naphthol pitch may be used in the manufacture of lacquers and for molding compositions.[95] Naphthylamine pitch is obtained as a by-product in the refining of naphthylamine.

Cresol Pitch. This is obtained as a residue in the distillation of crude cresol, and is known in Germany under the names "Karbolpech," "Kresolharz" and "Phenolpech." It has a fusing-point of 60 to 80° C., a decided cresol odor on heating and a brownish-black color. It is more or less soluble in aqueous caustic potash and almost completely soluble in alcohol and mixtures of alcohol and benzol.[96] It is similarly used in the manufacture of lacquers.

CHAPTER XVIII

WATER-GAS AND OIL-GAS TARS AND PITCHES

Water-gas tar, oil-gas tar and their corresponding pitches are not classified with "coal tar" and "coal-tar pitch," as they are intermediate in their properties between the latter and petroleum asphalts, on account of the petroleum products used in their manufacture. They are accordingly included in a separate chapter.

Carburetted Water-gas Tar. The mechanism of this process has already been briefly described. A modern water-gas plant hav-

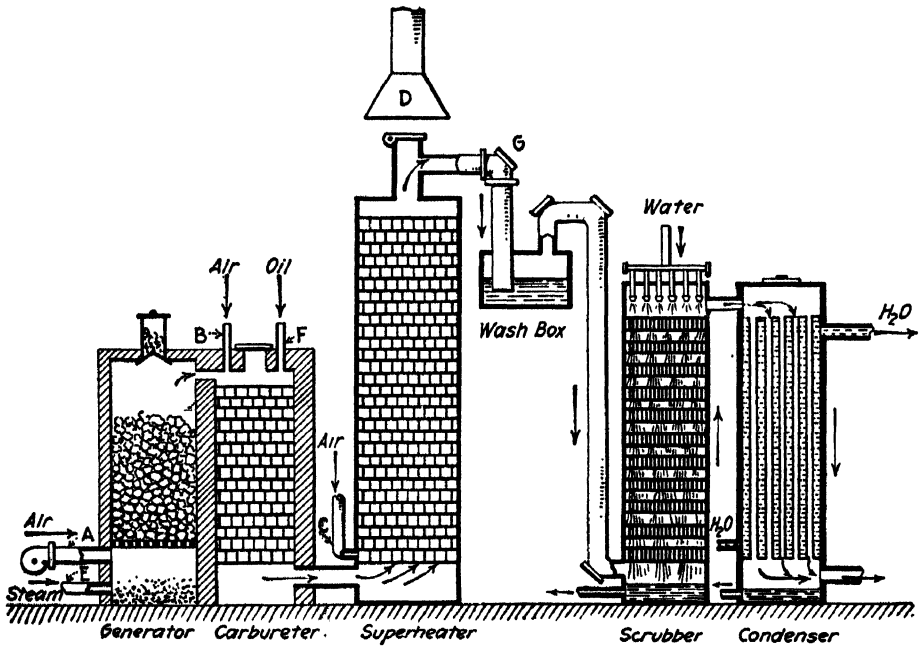


FIG. 117.—Lowe Water-gas Plant.

ing a capacity of 1½ to 3 million cubic feet of gas per day is illustrated diagrammatically in Fig. 117. This is known as the Lowe type of apparatus. Either anthracite coal or coke may be used as fuel. The fuel is charged into the generator and allowed to under-

go partial combustion by admitting a limited amount of primary air through the pipe *A* below the bed of fuel. The gases then pass downward through the carbureter and the combustion almost completed by means of a carefully regulated supply of secondary air introduced through the valve *B*. From the carbureter the products pass upward through the superheater, where the temperature may be controlled by admitting a tertiary supply of air through the valve *C*, and the products of combustion finally passed into the atmosphere through the stack *D*.

When the fuel in the gas-generator has been properly ignited, and the carbureter and superheater brought to the required temperatures, the air blasts are cut off in the sequence: *C*, *B*, and *A*, and the stack valve *D* closed. Steam is introduced into the generator through the valve *E* below the bed of incandescent fuel and results in the production of "blue-gas," according to the following reaction: $C + H_2O = CO + H_2$. This is passed into the carbureter where it mingles with a spray of carbureting oil consisting of "gas oil" or "fuel oil" introduced through *F*. The mixture is passed downward through the carbureter whereby the oil becomes vaporized. From the carbureter, the gases are passed up through the superheater, the temperature of which is very carefully regulated at 1200–1300° F. to crack the oil vapors into permanent gases, and this incidentally results in the formation of tarry matters.

The formation of carbon monoxide and hydrogen by the action of steam on incandescent fuel results in a lowering of the temperature, on account of the absorption or storing up of thermal energy, so that it becomes necessary to turn off the steam and reintroduce the air. The "blowing up" process is then repeated. At the same time the oil spray is turned off the carbureter, as its temperature has fallen to a point below which the oil would not be superheated sufficiently to convert it into a permanent gas. The "blowing" or "up run" lasts three to five minutes, and the "gas making" or "down run" lasts two to four minutes.

The water-gas and accompanying tarry vapors derived from the gas oil are passed from the superheater through the pipe *G* into a wash-box which corresponds to the hydraulic main in a coal-gas plant. The vaporization of water in the wash-box reduces the temperature of the gases from 1200 to 190° F. The gases next

pass upward through a scrubber and are then passed downward through a water-cooled condenser which reduces their temperature to 140–150° F., and thence into a relief gas-holder. Most of the tar is condensed in the wash-box and smaller quantities in the scrubber and condenser. An exhauster draws the gases through the foregoing train of apparatus and then forces them through a tar extractor, to remove the last traces of tar. The temperature of the gases as they pass through the tar extractor is in the neighborhood of 110–115° F. They are finally passed through the purifier filled with trays of ferric oxide to remove the sulfur compounds, and thence into the main gas holder.

The carbureting oil, known also as "gas oil" or "enriching oil" varies in composition, depending upon the character of the petroleum from which it is derived. Experience demonstrates that oils obtained from a paraffin-base petroleum generate the greatest proportion of gas and the smallest quantity of tar. Oils containing unsaturated straight-chain hydrocarbons are less efficient, and those containing unsaturated ring hydrocarbons are almost valueless. The yield of tar expressed in percentage by volume, based on the various types of petroleum used, is as follows:

Paraffin-base naphtha	2– 4 per cent
Paraffin-base gas oil	6–10 per cent
Paraffin-base crude oil	8–12 per cent
Asphaltic-base gas oil	10–15 per cent
Asphaltic-base crude oil	12–18 per cent

The quantity of carbureting oil ordinarily used varies from 2.5 to 4.5 gal. per 1000 cu. ft. of gas manufactured, depending upon the statutory requirements in the territory where the gas is distributed, as to its illuminating and heating values.

Properties of Water-Gas Tar. On account of the low specific gravity of water-gas tar, it readily forms an emulsion with the associated water and separates with great difficulty. The water thus retained may run as high as 85 per cent and the greater the percentage of water present, the more viscous will be the emulsion.[1] The water is practically free from ammonia compounds, thus differing from coal tar, and the tar is very much thinner in consistency, containing but a small amount of free carbon. The methods for dehydrating crude water-gas tar are similar to those used for coal

tars. The dehydrated water-gas tar complies with the following tests:

(Test 1)	Color in mass	Black
(Test 2*a*)	Homogeneity to the eye	Uniform
(Test 2*b*)	Homogeneity under microscope	Absence of carbonaceous matter
(Test 7)	Specific gravity at 77° F.	1.00–1.18
(Test 8)	Viscosity at 212° F. (100 cc.)	25–50
(Test 15*c*)	Fusing-point (cube method)	Less than 0° to 10° F.
(Test 16)	Volatile matter at 500° F., 5 hrs.	60–85 per cent
(Test 16*b*)	Distillation:	

		By weight	
Up to 110° C.	(Naphtha)	0 – 5	per cent (Sp. gr. 0.85–0.90)
110–170° C.	(Light oils)	0.5– 5.0	per cent (Sp. gr. 0.88–0.90)
170–235° C.	(Middle oils)	5 –35	per cent (Sp. gr. 0.98–1.00)
235–270° C.	(Heavy oils)	7 –30	per cent (Sp. gr. 1.00–1.07)
270–350° C.	(Anthracene oil)	10 –25	per cent (Sp. gr. 1.07–1.10)
Residue	(Water-gas-tar pitch)	20 –70	per cent

(Test 17*a*)	Flash-point	Low
(Test 19)	Fixed carbon	10–20 per cent
(Test 21)	Solubility in carbon disulfide	95 –100 per cent
	Non-mineral matter insoluble	0.2– 5 per cent
	Mineral matter	0 – ½ per cent
(Test 22)	Carbenes	0 – 2 per cent
(Test 23)	Solubility in 88° petroleum naphtha	20 – 75 per cent
(Test 26)	Carbon	90 – 95 per cent
(Test 27)	Hydrogen	3 – 6 per cent
(Test 28)	Sulfur	0.5–2.0 per cent
(Test 29)	Nitrogen	0.5–1.0 per cent
(Test 30)	Oxygen	1 – 2 per cent
(Test 32)	Naphthalene	Less than 10 per cent
(Test 33)	Solid paraffins	0 – 5 per cent
(Test 34*b*)	Sulfonation residue	1 – 25 per cent
(Test 37*e*)	Saponifiable constituents	Tr. – 2 per cent
(Test 39)	Diazo reaction	Yes
(Test 40)	Anthraquinone reaction	Yes

Water-gas tars consist principally of aromatic hydrocarbons and contain less naphthalene than coke-oven tars, negligible phenols and bases, also small amounts of solid paraffins and considerable naphthalenes. According to Downs and Dean,[2] water-gas tar contains substantial amounts of benzene, toluene, xylenes, naphthalene and anthracene. The nitrogenous bases and phenols are absent or nearly so. Weiss reports further that the percentage of free carbon varies from 1.04 to 1.087 per cent, with water-gas tars ranging in specific gravity from 1.078–1.090.[3] Water-gas tar may be vulcanized by heating with 5 to 8 per cent of sulfur.[4]

Oil-gas Tars. These are manufactured from petroleum alone without the use of coal or coke. Several methods have been used all embodying the same principle but differing in detail, the most important of which are as follows:

Pintsch Gas. This is manufactured by spraying gas-oil derived from petroleum in a closed retort constructed of iron or fire clay and heated to a temperature of 900 to 1000° C. by combustion of oil, gas or tar underneath. The shape of the retort is shown in Fig. 118. The vapors pass to the rear and thence downward and through a lower chamber into the hydraulic main in front. The gases are passed successively through a scrubber, condenser and purifier.

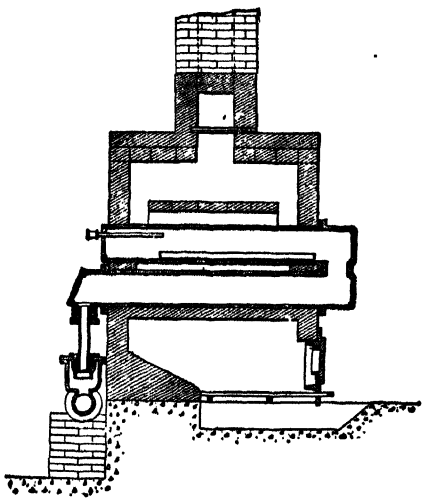


FIG. 118.—Pintsch Gas Retort.

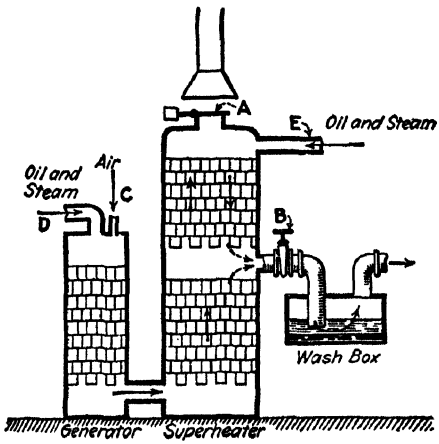


FIG. 119.—Oil-water Gas Plant.

Pintsch gas is used extensively for railroad and buoy lighting. It may be stored in holders under a pressure of 5 to 25 atmospheres, without suffering in illuminating power, as would prove to be the case with most other gases adapted for lighting purposes.

About 10 per cent tar is recovered in the Pintsch process, the characteristics of which will be described under the heading "oil-gas tar" below.

Oil-water Gas. This process is used almost exclusively on the Pacific coast for manufacturing illuminating gas, owing to the absence of coal deposits. The installation is shown diagrammatically in Fig. 119, and operates similar to a water-gas plant, except that petroleum is used instead of coal or coke.

It requires about 8 to 8½ gal. of fuel oil per 1000 cu. ft. of gas

(550–625 B.t.u.) of which about one-fifth is required for heating and four-fifths for gas-making. Higher temperatures are used for cracking than is the case in the manufacture of Pintsch gas or Blau gas, in consequence of which heavier tars are obtained, higher in free carbon and aromatics.

Oil-water-gas tar has also been termed "fuel-oil gas tar" and "reformed-gas tar." [5] At room temperatures it has a soft, semi-solid to almost solid consistency. Those produced at medium temperatures have a specific gravity at 60° F. of 1.15 to 1.20; insoluble in benzol 12 to 15 per cent; sulfonation residue 2 to 5 per cent. Those produced at high temperatures have a specific gravity at 60° F. of 1.30 to 1.35; insoluble in benzol 25 to 40 per cent; sulfonation residue less than 0.5 per cent.

Blau Gas. This is a further development of Pintsch gas, and is made by cracking oil vapors at a temperature lower than in the Pintsch process (i.e., 550 to 600° C.), but in a similar form of retort. The resulting gases are first purified by passing in the usual manner through hydraulic mains, coolers, cleaners and scrubbers to remove the tar, which amounts to 4–6 per cent of the oil used, and then compressed in a three- or four-stage compressor to 100 atmospheres, which causes the high boiling-point constituents to liquefy and absorb a large proportion of the non-liquefiable gases. The excess of the latter is used for running the compressor and heating the retorts.

The compressed Blau gas is so constituted that upon releasing the pressure, the dissolved and liquefied constituents are evolved in such proportions that the composition of the gaseous mixture remains constant. Blau gas is used principally for marine lighting purposes and is transported in cylinders of about 1 cu. ft. capacity, carrying 20 lb. of the compressed gas, which will expand to about 250 cu. ft. at atmospheric pressure. Its illuminating value is greater than that of Pintsch gas.

The tar recovered from the Blau gas process (about 15–20 per cent), is similar in its physical and chemical properties to the oil-gas tars described previously.[6]

Properties of Oil-gas Tars. Dehydrated oil-gas tars produced by the Pintsch process, the Blau gas process, and the oil-water gas process comply with the characteristics given on the following page.

Oil-gas tar may be vulcanized by heating with sulfur,[7] or by treating with sulfur dichloride.[8]

Refining of Water-gas and Oil-gas Tars. Oil-gas and water-gas tars when suitably dehydrated may be distilled in accordance with the methods used for "coal tar." Sometimes, either water-gas-

	Oil-gas Tars	
	Low Temperature (Pintsch- and Blau-gas Tars)	High Temperature (Oil-water-gas Tars)
(Test 1) Color in mass	Black	Black
(Test 2*a*) Homogeneity to the eye	Uniform	Gritty
(Test 2*b*) Homogeneity under microscope	Comparatively free from carbonaceous matter.	Contains considerable carbonaceous matter.
(Test 7) Specific gravity at 77° F.	0.95–1.10	1.15–1.35
(Test 8*a*) Viscosity at 212° F. (100 ml.)	25–50	Over 50
(Test 9*c*) Consistency at 77° F.	0	0 to 5
(Test 15*b*) Fusing-point (R. & B. method)	.....	30–100° F.
(Test 15*c*) Fusing-point (Cube method)	<0–20° F.	.....
(Test 16) Volatile at 500° F. in 5 hrs	35–70 per cent	25–50 per cent
(Test 16*b*) Distillation Test:		
Distillate to 315° C. (vol.)	30–75 per cent	20–50 per cent
Sp. gr. ditto at 60°/60° C.	.....	1.05–1.12
Residue at 315° C. (wt.)	25–70 per cent	50–80 per cent
Fusing-point ditto (R. & B. method)	.....	100–150° C.
(Test 17*a*) Flash-point	Low	Low
(Test 19) Fixed carbon	10–25 per cent	15–35 per cent
(Test 21) Soluble in carbon disulfide	99–100 per cent	70–90 per cent
Non-mineral matter insoluble	0–0.2 per cent	10–30 per cent
Mineral matter	0–0.5 per cent	0–0.5 per cent
(Test 22) Carbenes	0–2 per cent	0–2 per cent
(Test 23) Soluble in 88° petroleum naphtha	50–85 per cent	25–70 per cent
(Test 28) Sulfur	<1 per cent	< 1 per cent
(Test 30) Oxygen	1–2 per cent	1–2 per cent
(Test 32) Naphthalene	Trace	0–5 per cent
(Test 33) Solid paraffins	0–5 per cent	Trace
(Test 34*b*) Sulfonation residue	20–40 per cent	Trace to 10 per cent
(Test 37*e*) Saponifiable constituents	Trace	Trace
(Test 39) Diazo reaction	Yes (slight)	Yes (marked)
(Test 40) Anthraquinone reaction	Yes	Yes

tar pitch or oil-gas-tar pitch is mixed with coal-tar pitch in suitable proportions.

Properties of Water-gas-tar Pitch and Oil-gas-tar Pitch. Water-gar-tar and oil-gas-tar pitches may be distinguished from coal-tar pitches by:

(1) The small percentage of "free carbon" (non-mineral matter insoluble in carbon disulfide).

(2) The possible presence of paraffin wax (when non-asphaltic or semi-asphaltic petroleums are used).

On the other hand, water-gas-tar pitch may be distinguished from oil-gas-tar pitch by the following:

(1) Lower specific gravity of water-gas-tar pitch.

(2) Larger percentage of sulfonation residue from oil-gas-tar pitch. The sulfonated constituents are soluble in water, which serves to differentiate them from lignite- and shale-tars and pitches.

Water-gas-tar and oil-gas-tar pitches comply with the following tests:

	Water-gas-tar Pitch	Oil-gas-tar Pitch
(Test 1) Color in mass	Black	Black
(Test 2*a*) Homogeneity to the eye	Uniform	Uniform
(Test 2*b*) Homogeneity under the microscope	Small amount of carbon visible	
(Test 3) Aged surface	Variable	Variable
(Test 4) Fracture	Conchoidal	Conchoidal
(Test 5) Lustre	Bright	Bright
(Test 6) Streak	Black	Black
(Test 7) Specific gravity at 77° F	1.10–1.25	1.15–1.35
(Test 9*b*) Penetration at 77° F	0–100	0–100
(Test 9*d*) Susceptibility index	>100	>100
(Test 10) Ductility	Variable	Variable
(Test 11) Tensile strength at 77° F	Variable	Variable
(Test 15*a*) Fusing-point (K. and S. method)	80–275° F.	80–275° F.
(Test 15*b*) Fusing-point (R. and B. method)	100–300° F.	100–300° F.
(Test 15*c*) Fusing-point (cube method)	110–320° F.	110–320° F.
(Test 16) Volatile matter 500° F., 5 hrs. (per cent)	5–15	5–15
(Test 17*a*) Flash-point	300–400° F.	300–400° F.
(Test 19) Fixed carbon (per cent)	25–45	20–35
(Test 21) Solubility in carbon disulfide (per cent)	75–98	70–98
Non-mineral matter insoluble (per cent)	2–25	2–30
Mineral matter (per cent)	0–½	0–½
(Test 22) Carbenes (per cent)	5–10	5–10
(Test 23) Solubility in 88° petroleum naphtha (per cent)	50–70	60–80
(Test 28) Sulfur (per cent)	<4	<4
(Test 30) Oxygen (per cent)	0–2	0–2
(Test 33) Solid paraffins (per cent)	0–5	0–5
(Test 34*b*) Sulfonation residue (per cent)	0–15	20–40
(Test 37*e*) Saponifiable matter (per cent)	0–1	0–1
(Test 39) Diazo reaction	Yes (*)	Yes (*)
(Test 40) Anthraquinone reaction	Yes	Yes

* Slight—contain but a trace of phenols.

Both of these pitches are largely susceptible to changes in temperature, they are highly resistant to the prolonged action of moisture, and they are adapted for manufacturing low-priced solvent paints because of their ready solubility in "coal-tar naphtha."

CHAPTER XIX

FATTY-ACID PITCH, BONE TAR AND BONE-TAR PITCH

These are classified together because all are derived from substances containing animal or vegetable fats or oils, although in manufacturing bone tar and bone-tar pitch the crude materials carry but a small proportion.

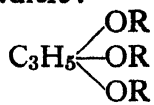
FATTY-ACID PITCH

Various generic terms have been used to designate this product, including candle tar, "Kerzenteer" (German) "goudron" (French), candle pitch, fat pitch and "Fettpech" (German). Specific names have also been applied, descriptive of the raw materials used in producing the pitch, such as stearin pitch, palm-oil pitch, bone-fat pitch, cotton-seed-oil pitch, cotton pitch, cotton-stearin pitch, cotton-seed-foots pitch, corn-oil pitch, corn-oil-foots pitch, packing-house pitch, garbage pitch, sewage pitch, fuller's-grease pitch, wool pitch, wool-grease pitch, wool-fat pitch, cholesterol pitch, cotton-oil pitch, cotton-seed-oil-foots pitch and stearin-wool pitch.

The fatty-acid pitches are obtained as by-products in the following manufacturing processes:

(1) Production of candle and soap stocks.
(2) Refining vegetable oils by means of alkalies.
(3) Refining refuse greases.
(4) Treatment of wool grease.

The raw materials used include the vegetable oils and fats, animal oils, fats and waxes (wool grease), also the waste greases derived from the foregoing. Vegetable and animal fats and oils are combinations of the fatty acids with glycerin, known as "triglycerides," and illustrated by the following generic formula, in which "R" represents any fatty acid radicle:

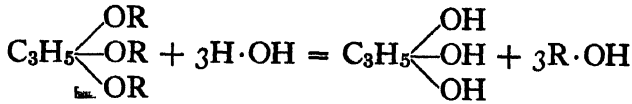


Fats and oils may be purified or refined in two ways:

(1) By treating vegetable oils with a small amount of caustic soda to remove the coloring matter, free fatty acids and other impurities, without, however, breaking up the triglycerides. This process is used for refining vegetable oils when they are to be used for edible purposes. The residue is treated with mineral acid to break up the soaps, and then distilled with steam to recover the fatty acids, whereupon a residue of fatty-acid pitch is obtained.

(2) By decomposing or "hydrolyzing" the triglycerides into glycerin and free fatty acids, and then distilling the latter with steam, whereby fatty-acid pitch is obtained as a residue. The object of distilling the fatty acids is to *improve their color or odor* and thereby adapt same (*a*) for the manufacture of candles (which are commonly light-colored or white), or (*b*) for manufacturing soaps (such as toilet soaps, etc.) which must be *odorless* and preferably light-colored.

Production of Candle and Soap Stocks. These are obtained from various animal and vegetable oils and fats, also from waste greases. It is *always* necessary to subject the fatty acids to a process of hydrolysis and steam distillation for producing candles, but not for manufacturing soaps, unless the fatty acids are too dark in color for the character of soap required or possess a disagreeable odor, in which event they are purified by distillation. Various methods of hydrolysis may be used, but they all depend upon the same reaction, in which the triglyceride combines with water and decomposes into glycerin and fatty acids, as illustrated in the following equation:



It is *necessary* to hydrolyze the fats or oils before distilling the fatty acids, since the triglycerides themselves are not capable of being distilled without decomposition. The following methods of hydrolysis have been used:

(*a*) *Hydrolysis by Means of Water.* Formerly, water alone was used for the purpose, the fat or oil being heated in an autoclave with 30 per cent of its weight of water at 220 lb. pressure (corresponding to a temperature of 200° C.) for eight to twelve hours.

This decomposes the triglyceride into fatty acids and glycerin, but with water alone it is difficult to break down the fat completely. It has been found that the addition of 3 per cent of lime or magnesium oxide, and preferably the latter, assists the reaction and produces a larger yield of a better product, and at a much lower temperature. Accordingly, the fat or oil is heated at 120 lb. pressure in a horizontal or vertical cylindrical vessel provided with a stirring device, with 20 to 25 per cent by weight of water and 3 per cent of lime or magnesium oxide. The breaking down of the fat is practically complete at the end of eight to ten hours, and in addition the color is very much better, as there is less decomposition, due to the lower temperature employed. The fats or oils used for this purpose may consist of animal or vegetable tallow, palm oil, bone fat, lard- or cotton-seed stearin (crystallized at low temperatures from lard or cotton-seed oil respectively), shea butter, etc.

At the end of the process, the free fatty acids rise to the surface and are skimmed off, leaving the aqueous liquor containing the glycerin (together with the hydrated lime or magnesia, when the latter are used). The glycerin is recovered by a special process which, however, does not fall within the scope of this treatise. The fatty acids are subjected to steam distillation to deodorize and whiten them, also to purify them by separating any non-hydrolyzed fat. The fatty acids are run into lead-lined tanks where they are first treated with dilute sulfuric acid to remove any traces of magnesium oxide, etc., then washed with water, heated to expel the moisture, after which they are fed into a retort and distilled with superheated steam with or without the use of vacuum. The fatty acids suitable for distillation should not contain more than 5 per cent of non-hydrolyzed fat (neutral fat) nor more than 0.2 per cent of mineral matter. To obtain a distillate of good quality, care should be taken not to distil the fatty acids at too high a temperature, as they are extremely susceptible to overheating and decomposition into dark-colored hydrocarbons (unsaponifiable), which would, of course, depreciate the value of the product.[1] The still should be constructed so that the flames will not come into direct contact with the bottom and cause local overheating. The temperature of the material in the still should preferably be maintained between 230 and 250° C., and although in certain instances it is permissible to reach a temperature of 270° C., under no circumstances should this be exceeded.

Two methods are used for conducting the distillation. The first consists in continuously replacing the fatty acids as they distil, with an equivalent quantity of undistilled material, as long as the distillate shows a satisfactory color and is free from unsaponifiable

hydrocarbons. The effect of the distillation is to concentrate the impurities and unsaponified (neutral) fats or oils in the still. A typical installation is illustrated in Fig. 120.[2]

The heating element in the still, *a*, consists of two copper coils of approximately 270 square feet (25 square meters) mean surface. Steam at about 450 pounds per square inch (32 kg. per sq. cm.) gage pressure is supplied by a motor-driven compressor, *b*. Vacuum is maintained at 7 to 10 mm. of mercury by use of steam ejectors, *c*. Approximately 10,000 pounds (4536 kg.) of crude fatty acid are charged to the still at the start of a run, and there-

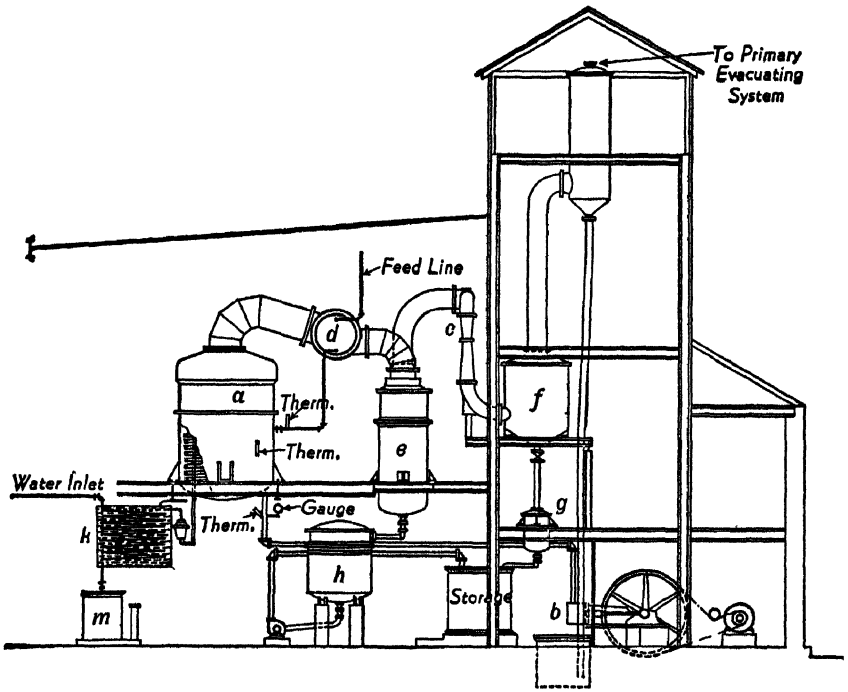


FIG. 120.—Distillation of Fatty Acids.

after for about 20 hours the feed is continuous to maintain an even level. The crude stock going to the still is preheated by passing through a heat exchanger, *d*, which also serves as a partial condenser for the fatty acid vapors. Most of the distillate is condensed in the jacketed coolers, *e*. A small amount of stock is condensed and baffled out in separator *f* and measured in receiver *g*. The bulk of the distillate is received in *h* and pumped out, under vacuum, to storage. The residue in the still *a* consists of soft fatty-acid pitch.

The second method consists in replacing the distilled fatty acids for but sixteen to twenty-four hours, then discontinuing the addi-

tion, and distilling the contents of the retort until the distillate ceases to be of suitable quality, as is evidenced by a change in its color. The residue consisting of soft fatty-acid pitch is then drawn off into a separate still known as the "pitch still," the first still recharged, and the process repeated, until after a sufficient number of distillations, a sufficient quantity of soft fatty-acid pitch accumulates for further treatment. It is claimed that the second method gives better results and yields a distillate lighter in color, containing a smaller percentage of hydrocarbons.

In either case the soft residue is distilled separately with superheated steam and vacuum. When the neutral fats increase in concentration to 12 to 15 per cent they commence to decompose into hydrocarbons, some of which distil with the fatty acids and some remaining with the residue. On distillation, the saturated fatty-acids pass over first and the residue contains an increasing proportion of unsaturated and hydroxy-acids, such as hydroxy-stearic acid, which at higher temperatures is converted into iso-oleic acid. At still higher temperatures the fatty acids are converted into hydro-carbons, which are principally unsaturated. The final residue constitutes the so-called "fatty-acid pitch" which amounts to between 2.5 and 5 per cent of the fatty-acids distilled.

(*b*) *Hydrolysis by Means of Concentrated Sulfuric Acid.*[3] The fats or oils are first freed from moisture by heating to a temperature of 120° C. It is essential that all the moisture be removed to prevent excessive decomposition. The mass is then *rapidly* mixed with 4 to 6 per cent of concentrated sulfuric acid (66 to 67° Bé.) and heated in a cylindrical vessel provided with a mechanical agitator. The heating is continued just long enough to break up the triglycerides and no longer. The sulfonated mass is then immediately run into boiling water and agitated by a steam jet until the sulfonated acids hydrolyze. The mass is then allowed to stand quietly until the free fatty acids rise to the surface, leaving the glycerol and sulfuric acid in the lower layer.

The fatty acids produced in this manner are dark colored and *must* be distilled. They are first washed with water until neutral, then heated to expel the moisture and finally distilled with superheated steam, with or without a vacuum as previously described, whereupon a residue of *soft fatty-acid pitch* is obtained. According to modern practice, this residue is again treated with concentrated sulfuric acid to hydrolyze any neutral fats remaining, and incidentally remove the accumulated mineral matter (including any copper or iron derived from the stills). It is then washed free from the acid and redistilled, leaving a residue of *medium* to *hard fatty-acid pitch.* The dark-colored distillate, known as "still returns,"

is worked up in small quantities with the crude fatty acids under going their first distillation.

The yield of stearin known in this case as "distillation stearin" is greater than that obtained in the aqueous process of hydrolysis, due to the fact that some of the olein (in this case known as "distillation olein" or "distilled olein") is converted into a solid product (consisting of stearolactone, isomeric oleic acid, etc.). The olein and stearin are separated by cooling, exactly as in the foregoing process. A smaller yield of glycerin is obtained due to its partial decomposition by the acid, and that of fatty-acid pitch is also less and of a darker color.

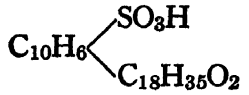
To avoid losing the glycerin, which constitutes one of the most important and highest priced products, a "mixed process" is now used consisting of a combination of the foregoing.

(*c*) *Hydrolysis by the "Mixed Process."* This is a combination of the two foregoing processes, and consists in first hydrolyzing the fats or oils in an autoclave with water and an alkaline accelerating agent (such as lime or magnesium oxide), and in this way recovering the full amount of glycerin. The resulting fatty acids are dehydrated and treated with concentrated sulfuric acid in accordance with process (*b*) to increase the yield of stearin and complete the hydrolysis of any neutral fat which may have escaped the first treatment, and thus minimize the formation of hydrocarbons in the distillate.

(*d*) *Hydrolysis by Means of Sulfo-compounds.* This process, known as the E. Twitchell method, is rapidly replacing the others, and is now employed in soap factories for treating the fats or oils before soap-making, as it separates a purer glycerin and at the same time results in a greater yield (88 to 90 per cent of the theoretical quantity contained in the fat or oil *vs.* 80 to 84 per cent obtained in the direct caustic soda saponification method for soaps). Moreover, the liquor separated in the Twitchell process is not contaminated with the sodium chloride used for "salting out" the soap in the ordinary method, and it contains 15 per cent by weight of glycerin against 3 to 4 per cent in the liquor obtained on direct saponification of the fats or oils with sodium hydroxide. The former therefore effects a saving in evaporation.

The fat or oil is first purified by steaming with 1 per cent of 60° Bé. sulfuric acid for about two hours. It is then transferred to a wooden vessel equipped with perforated steam pipes, also a well-fitting cover to exclude air which would cause the fatty acids to darken, and mixed with 50 per cent water and 1.5 per cent of the Twitchell reagent. The latter is prepared by allowing an excess of sulfuric acid to act on a solution of naphthalene (or other aro-

matic hydrocarbon) in oleic acid, which results in the production of a body having the general composition:



Naphthalene-sulfo-stearic acid.

It is advisable to introduce a small percentage of free fatty acids to start the hydrolysis which otherwise takes a little time to begin. The material is steamed for twenty-four hours, whereupon a small quantity (0.1 to 0.2 per cent) of 60° Bé. sulfuric acid is added to break up the emulsion and permit the fatty acids to rise to the surface and the glycerol to pass into the aqueous liquor below. About 0.05 per cent of barium carbonate is finally added to neutralize the mineral acid.

The resulting fatty acids are dark in color and must be distilled. This is usually affected after a preliminary treatment with concentrated sulfuric acid as in method (*b*) to increase the yield of stearin, which is of special importance when the product is to be used for manufacturing candles. The yield is the same as obtained from the saponification or mixed process respectively, depending upon the exact method of treatment.

(*e*) *Hydrolysis by Means of Ferments.* This method is also meeting with some favor, as it produces a large yield of glycerin uncontaminated with salt or other solids difficult of separation. Many soap manufacturers accordingly hydrolyze their stock by means of ferments to separate the glycerin, and then saponify the resulting fatty acids with sodium carbonate, either directly, or after first purifying them by steam distillation.

The ferment is derived from the castor plant by grinding the decorticated castor beans with water and filtering through cloth, whereupon a white creamy filtrate is obtained which is set to one side and allowed to ferment spontaneously. The ferment which rises to the surface is skimmed off and used while fresh. It is composed of a thick creamy substance containing approximately 38 per cent of fatty acids derived from castor oil, 58 per cent of water and 4 per cent of an albuminoid substance containing the active material.

The fat or oil to be treated is mixed with 40 per cent water, 5 to 8 per cent of the ferment and 0.2 per cent of manganese sulfate in a lead-lined vessel equipped with a steam coil and a perforated compressed-air pipe. Heat is then turned on, and the temperature maintained 2 to 3° C. above the melting-point of the fat or oil. The mass is agitated by air introduced through the perforated pipe and the treatment continued one to three days until the hydrolysis

is complete. Sufficient steam is then turned on to bring the mass to a temperature of 80 to 85° C., whereupon 0.30 to 0.45 per cent of 50 per cent of sulfuric acid is stirred in by air. This breaks up the emulsion, the clear fatty acids rising to the top and the aqueous liquor containing the glycerin settling to the bottom.

When the separated fatty acids are pale in color they may be saponified directly for manufacturing soaps. Where dark-colored fats, oils or greases have been employed, which result in the production of dark-colored fatty acids, the mass is distilled with steam, whereupon the fatty-acid pitch is obtained as residue. Candle stock may also be produced by subjecting the purified fatty acids to a low temperature and filtering, as described previously.

It is, of course, understood that when the crude oils or fats (triglycerides) or the free fatty acids derived from them (by any of the foregoing processes of hydrolysis) are saponified *directly* with sodium carbonate (soda ash), no fatty-acid pitch is produced.

Refining Vegetable Oils by Means of Alkali. Most vegetable oils intended for edible purposes, whether they are to be used for salad oils, lard substitutes, margarine manufacture, or directly for cooking oils and shortening, are first treated with caustic soda for the purpose of removing free acids, coloring matter, albuminous material, resins, etc. The oils chiefly treated are cotton-seed, corn, soya bean, cocoanut and peanut oils.

(*a*) *Refining Cotton-seed Oil.* Crude cotton-seed oil when obtained fresh from the seed varies in color from reddish brown to almost black. This is due in part to the coloring matter, which is a dark resinous substance capable of combining with caustic soda, forming a water-soluble salt, also albumin and pectin bodies. The method of refining the oil consists in agitating it with varying quantities of caustic soda solution, the strength of which will range from 1.10 to 1.20 specific gravity, according to the percentage of free fatty acids present and the practice of the individual refiner. The agitation is effected by mechanical stirrers in large tanks provided with heating coils. The quantity of alkaline liquor added is determined by careful laboratory tests and run in through perforated pipes. The effect of the alkali is first to darken the oil and apparently thicken it. After a short time small flakes begin to separate and heat is then applied. As the temperature increases, the flakes become larger, owing to the soap softening and running together. When the right point is reached, at temperatures varying from 100 to 130° F., steam and agitation are shut off and the soap drops to the bottom of the kettle, forming a mucilaginous mass, varying in

color from yellow to brown, through all shades of green and red. This material is known as the "foots." The clear light yellow oil which is pumped off the foots is then refined further for edible purposes. Cotton-seed oil purified in this manner is known to the trade as "summer yellow oil." When used in making lard substitutes it is bleached with fuller's earth and then deodorized, generally by the use of steam. Salad oil is obtained by chilling the summer yellow oil so as to crystallize out the palmitin which is separated by pressing or filtering.

In the United States alone the annual production of cotton-seed foots amounts to approximately half a million barrels. The foots vary in gravity from 0.97 to 1.04, averaging about 1.00. They contain the soda salts of the coloring matter, the soda soaps of any free fatty acids present in the cotton-seed oil (30 to 45 per cent), the coagulated albumin (8 to 12 per cent), phytosteryl, and varying quantities of mechanically entrained cotton-seed oil (triglycerides).

Cotton-seed foots are sold on the basis of "50 per cent fatty acid." As a matter of fact they contain between 35 and 65 per cent, averaging about 45 per cent. A representative sample contained:[4]

Fatty anhydrides (corresponding to a "50-per cent soap stock")	48.50 per cent
Glycerin	3.98 per cent
Caustic soda (Na_2O)	3.20 per cent
Foreign organic matter	5.90 per cent
Coloring matter	2.42 per cent
Water	36.00 per cent
Total	100.00 per cent

The cotton-seed foots may be converted directly into soap by boiling up with a small excess of caustic soda and "salting" it out in the usual manner, when no pitch will be obtained. The resulting soap is known as "killed foots" and the dark lye containing the coloring matter and impurities are run to waste. A process has also been described for recovering a shellac-like substance from cotton-seed foots by oxidizing with hydrogen peroxide in an alkaline solution and acidifying to separate the fatty matter.[5]

Usually, however, the cotton-seed foots are subjected to distillation. They are first boiled with sufficient alkali to complete the saponification and then treated with mineral acid to liberate the fatty acids. Or the foots may be acidified while hot with dilute sulfuric acid, whereupon a "black grease" containing about 90 per cent of the total fatty acids (calculated as oleic) rises to the surface. This is separated and subjected to the Twitchell or other hydrolyzing processes to break up any neutral fat and recover all

the glycerin. The fatty acids obtained in this manner are equivalent to 7.5 to 8.5 per cent of the original weight of the cotton-seed oil used. They are subjected to vacuum distillation with superheated steam to separate the pure fatty acids from the residue of fatty-acid pitch, variously called "cotton pitch," "cotton-oil pitch," "cotton-seed-oil pitch," "cotton-stearin pitch," "cotton-seed-oil-foots pitch," etc. The quantity of pitch produced will range between 10 and 20 per cent of the weight of the crude fatty acids (black grease) distilled, which is equivalent to 1 to 2 per cent by weight of the original cotton-seed oil. The yield will depend upon the degree the oil is saponified, the amount of impurities present, the efficiency of the distilling apparatus and the extent to which the distillation is carried. The still generally used holds 3 to 5 tons and the distillation is conducted intermittently. Two types of condensers are used, viz.: (1) water-cooled and (2) air-cooled. From these the vapors are passed through another condenser in which a water spray removes the lower fatty acids. Any volatile fatty acids distil over first, followed by the free fatty acids of higher boiling-points. The decomposition of any neutral fat present into unsaturated hydrocarbons and unsaturated fatty acids does not commence until the concentration of the neutral fat in the still reaches 12 to 15 per cent.

The purified fatty acids recovered by distillation are used for manufacturing soaps. The fatty-acid pitch is usually soft in consistency, moderately stringy and of a pale brown color when examined by transmitted light in thin layers.

(*b*) *Refining Corn Oil.* Corn oil is sometimes refined by treating with a small proportion of caustic soda in a manner similar to the method described for cotton-seed oil. Upon deodorizing the refined product with superheated steam under reduced pressure, while heated to a temperature of 400° F., an edible product is obtained, used as a salad oil, also for cake and biscuit making. It may also be converted into a lard compound by a hydrogenation process. The corn-oil foots are treated by a method similar to the one used for refining cotton-seed foots. A pitch is obtained known as "corn-oil pitch," possessing a comparatively high fusing-point, characterized by its rubber-like properties and lack of ductility. If the distillation is carried too far, the pitch will actually solidify in the still and can only be removed with great difficulty.

Refining Refuse Greases. (*a*) *Refining Packing-house and Carcass-rendering Greases.* "Tallow" is the name applied to the purified solid fat or "suet" obtained from cattle. It is used extensively for producing soap and candle stock. The crude fat is first "rendered" by boiling with water in an open vessel to separate

it from any albuminous matter or other impurities present, and then clarified by washing with weak brine. "Lard" is obtained by rendering the soft fats which surround the kidneys, intestines and backs of pigs. Tallow and lard may be used as such for manufacturing soaps, but for producing candles they must first be hydrolyzed and purified by steam-distillation.

The waste meat scraps obtained from packing houses, also the carcasses of animals freed from the bones, are treated with steam in large digestors at a high pressure to separate the fat. When the cooking is complete, the batch is allowed to stand quietly to permit the grease to rise to the surface and the disintegrated meat-fibers to settle. The grease is skimmed off and mixed with any additional grease recovered from the settlings by filter-pressing. The residue is then converted into fertilizer, and the aqueous liquid used for making glue. The grease recovered from this process has a disagreeable odor and a dark color, and must be hydrolyzed and steam-distilled before it can be used for manufacturing either candles or soap. The residue from the steam distillation process, amounting to between 5 and 6 per cent of the grease, constitutes a variety of fatty-acid pitch having a light brown color when viewed in a thin layer and great ductility (unless the pitch is distilled too far). A packing-house grease has been extensively marketed in this country under the name of "yellow grease."

(*b*) *Refining Bone Grease.* The bones recovered from packing houses or carcass-rendering works are used for manufacturing glue, bone-black (used for decolorizing petroleum distillates), and fertilizer. Bones from the head, ribs and shoulder-blades contain 12 to 13 per cent of fat, whereas the large thigh bones ("marrows") contain 20 per cent. The fat is extracted by breaking up the bones into small fragments and then either:

(1) Treating with steam in an autoclave under a pressure of 2 to 3 atmospheres, whereupon a portion of the fat separates and floats to the surface, the gelatin or glue goes into solution, and the mineral ingredients (calcium phosphate, etc.) remain as residue. From 8 to 9 per cent of fat (based on the dry weight of the bones) is recovered in this manner.

(2) Extracting the dried bones with a volatile solvent such as naphtha, carbon tetrachloride, or benzol in a suitable apparatus. A much higher percentage of fat is extracted in this manner, but the cost of operation is higher, due to unavoidable losses of solvent, and the odor of the product is very strong.

In either event the extracted bone fat is first hydrolyzed by any of the foregoing methods and then steam-distilled, whereupon a variety of fatty-acid pitch, known as "bone-fat pitch," is recovered as residue, amounting to 5 to 6 per cent by weight of the bone-fat.

The product may be used for manufacturing soap, or after cooling and filtering, the "stearin" may be converted into candles, and the "olein" either used for manufacturing soap or else marketed as such for "wool oils."

(*c*) *Refining Garbage and Sewage Greases.* The average city garbage as collected contains:

Water	70–80 per cent
Grease	3– 4 per cent
Tankage	10–20 per cent
Tailings (rubbish)	3– 6 per cent

It is treated in a manner similar to that used for working up the refuse from packing houses and carcass-rendering establishments, namely by boiling in large digestors holding 8 tons for six hours under a pressure of 70 to 80 lb. (Arnold-Egerton System).[6] This reduces the material to a pulpy mass, which is filter-pressed to remove the water and grease. The residue, known as "tankage," is dried and ground for use as fertilizer. The filtrate is allowed to stand, whereupon the grease rises to the surface and is skimmed off. The grease, when dehydrated, has a dark brown color. It is hydrolyzed to separate the glycerin, and the resulting fatty acids purified by steam-distillation to render them suitable for manufacturing soaps and candles. The residue, known as "garbage pitch," amounting to 5 to 7 per cent by weight of the garbage, has a dark color when viewed in a thin layer, and is quite susceptible to temperature changes.

Sewage also carries a proportion of grease which is now often being recovered, especially in large cities. The sewage is first run into large tanks, where the solid matter known as "sludge" settles to the bottom. The precipitation may be accelerated by adding a small percentage of slaked lime. After drawing off the liquor, the sludge is treated with a small quantity of sulfuric acid to break up any insoluble soaps, and then boiled in large digestors under pressure to hydrolyze the fats, and enable the grease to separate. The residue is dried and used as fertilizer. The grease is dehydrated, then hydrolyzed and finally distilled with superheated steam, yielding about 25 per cent of a fatty-acid pitch known as "sewage pitch." The characteristics of this are similar to those of garbage pitch. The distillate contains about 50 per cent of liquid olein and 50 per cent of solid stearin melting at about 113° F.[7]

(*d*) *Refining Woolen-mill Waste.* Olive oil, lard oil, neat's-foot oil, saponification olein (or saponified olein) and distillation olein (or distilled olein) are sold under the names "wool oils" or "cloth oils," and used in woolen mills for lubricating the wool before spinning into yarn, or for oiling old woolen rags before grind-

ing and "pulling" in the manufacture of "shoddy." One of the perquisites of the wool oils is that they shall have no tendency to dry or oxidize, and they must also be easily removable on boiling the finished woolen goods or shoddy with a solution of soap or sodium carbonate. The presence of hydrocarbons, even in small quantities, is objectionable, as they tend to prevent the removal of the wool oils during the scouring process.

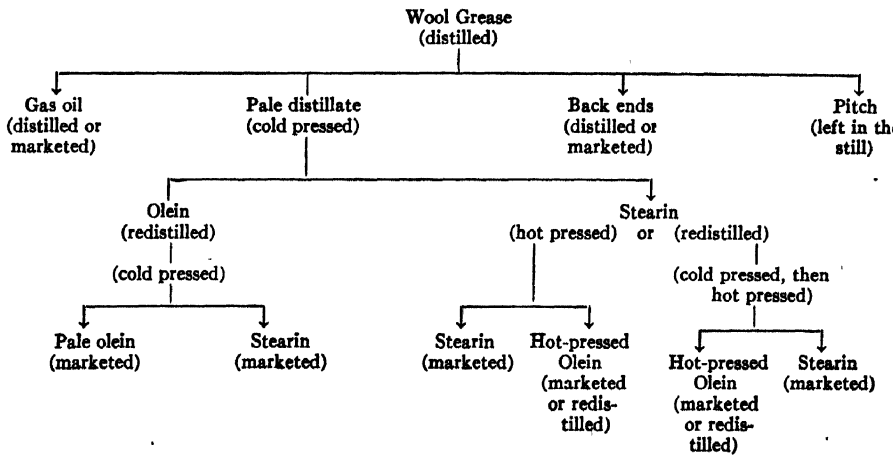
After the goods are scoured, the liquid is mixed with freshly slaked lime which serves to precipitate the soaps. The curds are settled out and separated, then acidified with dilute sulfuric acid to separate the free fatty acids, which are skimmed off and filtered to remove any dirt. The product known as "fuller's grease," "seek oil," or "magma oil," is distilled with steam, whereby the wool oils are recovered, and a residue of fatty-acid pitch obtained, amounting to 10 per cent by weight of the grease; known as "fuller's-grease pitch," or "seek-oil pitch." This will vary in its properties depending upon the raw materials entering into the composition of the original wool oils.

Treatment of Wool Grease. Wool grease, known also as "wool wax," or "wool dégras," represents the oily material naturally present in sheep's wool, and differs entirely from the so-called "wool oil" discussed previously. Wool grease is in reality an animal wax, as it contains no glycerin or glycerides whatsoever. It is extracted by boiling the cut wool with an alkaline soap solution or sodium carbonate. Formerly, volatile solvents were used for this purpose, but the method is no longer practiced. After boiling with soap or sodium carbonate, the liquor is acidified with sulfuric acid, whereupon grease rises to the surface and is skimmed off. Dehydrated wool grease melts between 86 and 104° F. and contains approximately 55–60 per cent fatty acids, also 40–45 per cent higher alcohols (unsaponifiable).

Wool grease is treated in various ways, and among others by a direct process of distillation with superheated steam *without* previous hydrolysis (as the waxes present are not amenable to such treatment) whereby the following reactions take place:[8] (1) distillation of the free fatty acids, which continues up to one-third of the distillate; (2) decomposition of free hydroxy acids principally into lactones, some of which distil over and some decomposing into carbon dioxide and solid unsaturated acids; (3) decomposition of neutral esters into unsaturated acids and unsaturated hydrocarbons.

Since wool grease contains an average of 65 per cent esters, this will account for the high percentage of unsaponifiable matter in wool grease products; (4) distillation of hydrocarbons. About 39 per cent of the total fatty acids in wool grease is oxidized acids, and of these about 25 per cent work their way into the distillate and the remainder are found in the residual pitch. The pitch consists of about 85 per cent polymerized hydrocarbons and 12 to 15 per cent fatty acids (of which 40 to 65 per cent are oxidized acids). The course of the treatment is shown in Table XXVII. The residue of fatty-acid pitch is known as "wool-grease pitch," "wool-fat pitch," "wool pitch," or "cholesterol pitch." The olein (known as "distilled-grease olein" or "dégras oil"), is used as a leather or wool oil, and the stearin (known as "dégras stearin"), is used in the soap industry or as a leather "stuffing grease."

TABLE XXVII



Physical and Chemical Properties of Fatty-acid Pitches. Fatty-acid pitches vary considerably in their physical and chemical properties, depending upon the following circumstances:[9]

(1) The nature of the fat or oil from which the fatty acids are derived. If these contain low melting-point fatty acids, the fatty-acid pitch will be soft in consistency, provided the distillation has not been carried too far. On the other hand, if high melting-point fatty acids predominate, the fatty-acid pitch will be semi-solid to solid in consistency.

(2) The proportion of neutral fats or oils present in the fatty-

acid mixture, which will prove substantial if the process of hydrolysis is not carried to theoretical completion. Since the neutral fats or oils (triglycerides) do not distil with superheated steam, they concentrate in the fatty-acid pitch, and are very apt to decompose into hydrocarbons (unsaponifiable) if the distillation is carried too far. If the distillation is stopped at a point without decomposing the neutral fats or oils, the value of the fatty-acid pitch is enhanced by their presence, as they are more stable and weather-resistant than the fatty acids themselves.

(3) The extent to which the distillation is carried and the temperature at which it is performed. If distilled too far or at too high a temperature, the fatty acids decompose in the presence of steam, first into hydroxy acids, which in turn break down into lactides, unsaturated products and lactones in the following manner:

(*a*) α-hydroxy acids are converted into lactides.

(*b*) β-hydroxy acids become converted into unsaturated products.

(*c*) γ and δ hydroxy acids are transposed into cyclic esters of the nature of γ and δ lactones respectively.

It follows, therefore, that the fatty-acid pitches contain free fatty acids (mostly polymerized), their lactones (anhydrides), undecomposed glycerides (neutral fats or oils), condensation products of unknown composition, hydrocarbon decomposition products, and in the case of fatty-acid pitches derived from wool grease, we find cholesterol and higher alcohols.[10] Their cryoscopic molecular weights vary from 370 to 680, and the so-called "rubber pitches" show the highest molecular weights and contain up to 80 per cent saponifiable matter.

The presence of hydrocarbon decomposition products is evidenced largely by the color of the pitch when examined in a thin layer. If these are present, the pitch will be a black, otherwise it will have a rich brown color. The percentage of saponifiable constituents present in the fatty-acid pitch is a criterion of its quality. The larger the percentage, the better will be the quality from the standpoint of weather-resistance. Fatty-acid pitches of the optimum quality contain not less than 90 per cent of saponifiable constituents. *They are as weather resistant as any bituminous substance.* The smaller the percentage of saponifiable constituents in the pitch, the less weather-resistant it will prove to be.

In recent years there has been a tendency to remove more and

more of the saponifiable ingredients from the fatty-acid pitches, in view of the high price commanded by the fatty acids, and also because of improvements effected in the distillation process. The author has examined fatty-acid pitches containing as high as 98 per cent unsaponifiable constituents. These appear glossy black in color and almost opaque in a thin layer, and therefore find a ready use in the manufacture of cheap lacquers and japans, not intended for exposure out of doors.

All fatty-acid pitches are converted in a more or less *infusible* and *insoluble* mass upon exposure to the weather for a long period, or upon heating a short time to a temperature of 250 to 350° C. in contact with air.[11] This is equally true whether or not unsaponifiable constituents are present, and makes this class of pitches especially valuable for manufacturing, baking japans and varnishes.[12] They may also be hardened, or converted into insoluble and infusible substances by heating with sulfur,[13] or with sulfur and an alkaline "accelerator" (e. g., thiocarbanilide or diphenylguanidine),[14] or by heating a mixture of fatty-acid pitch and phenol or cresol with sulfur dichloride or selenium monochloride,[15] or by heating a mixture of fatty-acid pitch and phenol or cresol with formaldehyde or hexamethylenetetramine which results in the formation of a hard resinous condensation product.[16]

Various methods have been proposed for blowing fatty-acid pitches, including: blowing wool-fat pitch with air at an elevated temperature;[17] blowing a mixture of fatty-acid pitch and montan wax, with or without the addition of colored mineral pigments;[18] etc. Blowing with air at elevated temperatures rapidly increases their fusing-point and at the same time tends to convert them into the insoluble modification.[19]

The insoluble (polymerized) form of fatty-acid pitch may again be rendered soluble in organic solvents by a process of mechanical mixing (i. e., "hot-rolling") with natural resins, resin esters, or synthetic resins.[20]

A highly elastic form, termed "rubber pitch," is obtained by heating soft fatty-acid pitch to 240–250° C., with 10 per cent of either sulfuric or nitric acid,[21] and the resultant product is characterized by being extremely resistant to temperature changes, approaching vulcanized rubber in its physical properties. Soft pitches

may also be hardened by heating with oxides of magnesium, manganese or lead; lime,[22] picric acid,[23] etc.

Fatty-acid pitches containing a large proportion of saponifiable constituents show an extremely *low* susceptibility index, in fact lower than any other class of bituminous materials. Conversely fatty-acid pitches in which the unsaponifiable constituents predominate are apt to have quite a high susceptibility index.

In general, the various classes of fatty-acid pitch are characterized by the following predominating *physical* properties, assuming that they have been carefully prepared and neither overheated nor distilled too far, viz.:

Fatty-acid pitches made from lard are usually very ductile with a low susceptibility index.
Fatty-acid pitches made from tallow are generally hard, lacking in ductility with a low susceptibility index.
Palm oil pitches are hard, lacking in ductility with a moderately high susceptibility index.
Cotton-seed-foots pitch is usually soft, of moderate ductility having a low susceptibility index.
Corn-oil-foots pitch is *extremely* rubbery, shows little ductility and has an *extremely* low susceptibility index.
Packing-house pitch is ductile and has a low susceptibility index.
Bone-fat pitch lacks ductility and has a moderately high susceptibility index. Its color in a thin layer and streak are black.
Garbage and sewage pitches are ductile with a high susceptibility index. Their color in a thin layer and streak are usually black.
Wool-grease pitch is ductile and has an extremely high susceptibility index. Its color in a thin layer and streak are usually black.

Fatty-acid pitches (referring to all types) comply with the following characteristics:

(Test 1)	Color in mass	Dark brown to black
(Test 1*a*)	Homogeneity to the eye at 77° F.	Uniform to gritty
(Test 2*b*)	Homogeneity under miscroscope	Uniform to lumpy
(Test 3)	Appearance surface aged indoors one week.	Bright
(Test 4)	Fracture	None to conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak on porcelain	Light yellow, brown to black
(Test 7)	Specific gravity at 77° F.	0.90–1.10
(Test 9*b*)	Penetration at 77° F.	8 to above 360
(Test 9*c*)	Consistency at 77° F.	0– 40
(Test 9*d*)	Susceptibility index	8– 40
(Test 10)	Ductility	Variable

(Test 15*a*)	Fusing-point (K. and S. method)	35–225° F.
(Test 15*b*)	Fusing-point (R. and B. method)	50–245° F.
(Test 16)	Volatile matter 500° F., 5 hours	0.5–7.5 per cent (*See Note*)
(Test 17*a*)	Flash-point	450–650° F.
(Test 19)	Fixed carbon	5– 35 per cent
(Test 21)	Solubility in carbon disulfide	95–100 per cent
	Non-mineral matter insoluble	0– 5 per cent
	Mineral matter	0– 5 per cent
(Test 22)	Carbenes	0– 5 per cent
(Test 23)	Solubility in 88° petroleum naphtha	80–100 per cent
(Test 28)	Sulfur	0 per cent
(Test 30)	Oxygen	2– 10 per cent
(Test 33)	Solid paraffins	Trace
(Test 34*a*)	Saturated hydrocarbons	0– 5 per cent
(Test 34*b*)	Sulfonation residue	0– 5 per cent
(Test 37*a*)	Acid value (including lactone value)	2–100
(Test 37*c*)	Ester value	40–125
(Test 37*d*)	Saponification value	60–200
(Test 37*e*)	Saponifiable constituents	5– 98 per cent
	Unsaponifiable constituents	2– 95 per cent
(Test 37*f*)	Hydrocarbons in unsaponifiable matter	90–100 per cent
	Higher alcohols (cholesterol) in unsaponifiable matter	0–10 per cent
(Test 37*b*)	Glycerol	Tr.–2.5 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No
(Test 41)	Liebermann-Storch reaction	Yes in the case of the wool grease pitches *only*

NOTE. In nearly all cases, a skin will form over the surface of the pitch during the determination of the volatile matter. This is characteristic. Certain fatty-acid pitches, especially those containing a large percentage of saponifiable constituents, often toughen up and solidify to a rubber-like mass during this test.

Table XXVIII contains the results of the examination of representative samples of fatty-acid pitch by the author.

According to Julius Marcusson [24] the saponification value of fatty-acid pitches ranges from 24 to 106, whereas the saponification value of petroleum asphalts does not exceed 21. Lukens [25] reports that the saponification value of fatty-acid pitches ranges from 45 to 100 and of petroleum asphalts from 5 to 18.

Fatty-acid pitches may be distinguished from asphalts by the small percentages of sulfonation residue and sulfur. Marcusson contends that fatty-acid pitches contain a trace of sulfur compounds, derived from impurities present in the original fats and also from the sulfuric acid used for hydrolysis. Such sulfur compounds are distinguished from those occurring in asphalts by the fact that they are not precipitated by mercuric bromide (Test 28). It is also interesting to note in this connection that no such precipitate is obtained from fatty-acid pitches that have been vulcanized with sulfur.

TABLE XXVIII.—CHARACTERISTICS

No.	Test	From Lard				From Tallow	From Packing-house Refuse
	Physical Characteristics:						
2*a*	Homogeneity to eye at 77° F.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.
2*b*	Homogeneity under microscope	Homo.	Homo.	Lumpy	Homo.	Homo.	Homo.
3	Appearance surface aged seven days	Bright	Bright	Bright	Bright	Bright	Bright
4	Fracture	.....	.....	.....	.....	Conch.	.....
5	Lustre	.....	.....	.....	.....	Bright	.....
6	Streak	Yellow	Yellow	Yellow	Brown	Brown	Brown
7	Specific gravity at 77° F.	0.972	0.980	0.990	1.000	1 060	1.003
	Mechanical Tests:						
9*b*	Penetration at 115° F.	Soft	Soft	Soft	220	36	Soft
	Penetration at 77° F.	Soft	Soft	280	99	21	100
	Penetration at 32° F.	88	85	83	69	16	68
9*c*	Consistency at 115° F.	0.0	0 0	0.0	1.5	11.3	0.6
	Consistency at 77° F.	0.7	1.3	2.4	5 8	23 2	6 3
	Consistency at 32° F.	8.2	9.8	10.0	15 2	52.0	15.7
9*d*	Susceptibility index	18.3	16.7	14.3	12.5	22 3	15.6
10*b*	Ductility in cms. at 115° F.	0	0	10	2.5	18 5	23
	Ductility in cms. at 77° F.	42	36	25.5	28 5	4	81 5
	Ductility in cms. at 32° F.	75	88	27	18	0	20
11	Tensile strength in kg. at 115° F.	0.0	0.0	0.0	0 05	1.75	0.0
	Tensile strength in kg. at 77° F.	0.0	0.05	0.05	0.20	3 10	0 40
	Tensile strength in kg. at 32° F.	0 75	0 95	1 75	2 35	6 2	3.25
	Thermal Tests:						
15*a*	Fusing-point, deg. F. (K. and S. method)	44.5	59	70	110	182	97
15*b*	Fusing-point, deg. F. (R. and B. method)	57	74	85	130	202	115
16	Volatile 500° F. in 5 hours, per cent.	2.6	3 0	5.3	5.0	1.2	1.7
	Character of residue	Little Change	Little Change	Gelat.	Little Change	Little Change	Little Change
17*a*	Flash-point, deg. F.	452	525	485.5	486	482	510
19	Fixed carbon, per cent.	6.2	11.2	10 4	12.0	18.4	12.6
	Solubility Tests:						
21	Soluble in carbon disulfide	100.0	100.0	99.7	100.0	98.5	100.0
	Non-mineral matter insoluble	0.0	0.0	0.3	0.0	2.1	0.0
	Mineral matter	0.4	1.4	1.5	0.5	1.3	2.0
22	Carbenes	0.0	0.0	0.0	0.0	0.1	0 0
23	Soluble in 88° petroleum naphtha	100 0	100.0	100.0	98.0	86.7	100.0
	Chemical Tests						
28	Sulfur	0.0	.....	.....	.....	0.0	.....
33	Solid paraffins	0.0	0.0	0.0	0.0	0.0	0.0
34*b*	Sulfonation residue	0.0	0.3	0.0	.....	.....	3.7
37*a*	Acid value	17.6	65.0	63.3	81.95	44.1	23.5
37*b*	Lactone value	63.2	79.8	75.9	97.55	112.0	74.1
37*c*	Ester value	86.4					
37*d*	Saponification value	167.2	144.8	139.2	179.5	156.1	97.6
37*e*	Unsaponifiable matter	0.5	5.5	1.5	1.0	15.2	5.0
37*f*	Hydrocarbons in unsaponifiable matter	.....	95.0	.....	.....	97.0	.....
37*f*	Higher alcohols in unsaponifiable matter	.....	5.0	.....	.....	3.0	.....
37*h*	Gycerol	4.7	.....	.....	.....	0.0	.....

OF TYPICAL FATTY-ACID PITCHES

From Bone-fat	From Garbage	From Sewage	From Cottonseed-oil Foots		From Corn-Oil Foots	From Palm-oil		From Wool-grease
Homo. Gritty Bright .... Black 1.036	Homo. Gritty Bright .... Black 1 063	Gritty Lumpy Bright .... Black 1.011	Homo. Lumpy Bright .... Yellow 0.992	Homo. Homo. Bright .... Brown 0.955	Gritty Lumpy Bright .... Brown 0 998	Homo. Homo. Dull Conch. Sl. dull Brown 1.042	Homo. Gritty Bright Conch. Bright Black 1.087	Gritty Lumpy Bright .... Black 1.020
150	145	130	Soft	260	61	46	35	Soft
76	50	14	200	120	34	19	10	150
14	12	18	72	66	46	14	5	34
2.4	2.6	2.9	0.0	1.1	7 2	9 3	11.7	0.0
7.9	11.7	30.0	2.7	5.1	15.8	24.7	35.2	4.2
55.4	60.1	48.3	14.1	16.5	25.1	56.5	82 0	32.8
41.9	40 5	33.9	19.7	15.3	8.5	29.5	41.0	35.8
18	72.5	52	8.5	31	5	35.5	25	15
5	60	15	19	26.5	2	2	0.5	47.5
0	0	0.5	28.5	10	0.5	0	0	31
0.05	0.50	0.25	0 0	0 05	0.75	1.00	2.20	0.0
0 30	1.25	1 10	0.10	0.5	1.55	2.2	5.00	0.25
4 70	6 2	8.0	2 30	4.2	5.4	9.5	11.25	1.05
126.5	142	134	71.5	101	210	161.5	172	91.5
144	162	151.5	92	120	235 5	186	193	108 5
0 5	0.6	0.55	4.2	0.38	0.25	3.5	1.2	7.2
Little Change	Little Change	Gelat.	Gelat.	Gelat.	Gelat.	Much Change	Little Change	Little Change
575	590	540	635	525	580	462	504	460
19.4	33.5	18.3	10.8	9.2	8.2	26.2	34.0	30.6
97.7	98.2	98.2	99.8	97.5	97.3	98.2	96.2	98.8
0.0	0.7	1.7	0.2	0.4	2.0	0.8	3.8	0.5
0.3	0.35	0.8	1.4	0.3	0.5	1.2	1.3	1.1
0.1	0.4	1.1	0.0	0.0	0.2	2.1	4.3	0.0
94.6	88 3	95.2	100.0	92.1	96.4	92.0	82.2	99.0
0.0	0.0	.....	.....	0.0	.....	0 0	.....	.....
0.0	0.0	0.1	0.0	0.0	0.0	0.2	0.3	0.0
4.0	.....	3.0	0.0	.....	0.5	1.2	.....	.....
20.2	2.5	.....	60.5	.....	.....	46.5	13.4	36.5
39.5	103.5	.....	95.4	.....	.....	92.9	53.9	88.7
59.7	106.0	120.4	155.9	151.0	172.0	139.4	67.3	125.2
98.0	97.0	7.0	2.0	4.6	2.7	6.0	75.0	60.6
93.6	91.7	90.5	95.0	.....	.....	96.0	.....	90.0
6.4	8.3	9.5	5.0	.....	.....	4.0	.....	10.0
.....	.....	0.55	0.8	2.35	.....	.....	.....	.....

Only cyclic bodies containing sulfur in ring formation give precipitates with mercury.

E. Donath and B. M. Margosches[26] state that wool-grease pitch may be identified by boiling 10 g. of the material with 50 cc. N–2 alcoholic potash under a reflux condenser for ½ hour, and then filtering the hot liquid. A precipitate will form in the filtrate on cooling, consisting of the potassium salts of hydroxy fatty acids, which upon recrystallizing from boiling 95 per cent alcohol and transposing with hydrochloric acid, will form a white crystalline mass of the hydroxy fatty acids melting at 80–82° C., which is insoluble in water and soluble in hot naphtha. It is claimed that by this method 25 per cent of wool-grease pitch may be detected in admixture with other pitches. A further means of identification depends upon the fact that wool-fat pitches give the cholesterol reaction.

The author has found the lineal coefficient of expansion of fatty-acid pitches for 1° F. (length = 1) to average 0.00023.

Fatty-acid pitches flux satisfactorily with mineral waxes, native and pyrogenous asphalts, tars and pitches. They also flux satisfactorily with gilsonite and glance pitch, but *not* with grahamite.

The following figures indicate that the hardening (toughening) of fatty-acid pitches on exposure to the weather is due to *oxidation*. A sample of soft fatty-acid pitch (the first in Table XXVIII) was melted and poured into a shallow glass dish, forming a layer *exactly* 1 millimeter thick. This was exposed out of doors for one year in a dust-free receptacle, protected from the direct action of the weather, and the following increases in weight noted:

After 1 month gained	0.62 per cent	After 7 months gained	4.20 per cent
2 months	2.52 per cent	8	4.27 per cent
3	3.27 per cent	9	4.30 per cent
4	3.50 per cent	10	4.38 per cent
5	3.86 per cent	11	4.42 per cent
6	4.12 per cent	12	4.46 per cent

The original pitch was soft and semi-liquid, but after exposure for one year it hardened to a tough, leathery mass. The original fusing-point was 44½° F. (K. and S. method), and at the end of the year it was 185° F.

BONE TAR AND BONE-TAR PITCH

In the production of bone tar and bone-tar pitch the crude bones are first steeped in a 1 per cent solution of brine for three to four days, to separate the fibrous matter. They are then degreased by one of the following methods:

(1) Boiling the bones with water in open vessels;
(2) Boiling with water in closed tanks under a pressure of 10 lb.;
(3) Extracting the bones with a solvent (usually a petroleum distillate boiling at 100° C.) and removing the last traces of solvent from the bones by blowing in live steam. The degreased bones are then treated to extract the glue by again subjecting the bones to the action of live steam for a lengthy period under a pressure of 15 to 20 lb. in an upright cylindrical boiler with a false bottom. The glue gradually leaches from the bones, the quantity extracted depending upon the duration of the treatment. When it is desired to convert the bones into "bone charcoal" (used for the purification of petroleum distillates and paraffin wax), only one-half of the gelatinous matter is extracted from the degreased bones, whereupon the water is drained off and the bones allowed to air-dry. They are then distilled destructively in horizontal cast-iron retorts, the distillate being condensed, and the permanent gases consumed under the retort. The distillate consists of an alkaline aqueous liquor containing ammoniacal bodies, and 1.5–2.0 per cent (based on the weight of raw material used) of a tarry layer known as "bone tar," "bone oil," "Dippel oil," or "Oleum animale foetidum." The residue remaining in the retort, known as "bone charcoal" or "animal charcoal," is removed while still hot, and transferred to an air-tight vessel in which it is allowed to cool. It is then passed through grinding mills and sieved. The bone charcoal is composed of approximately 10 per cent of carbon, 75 per cent of calcium phosphate, the balance consisting of various other mineral ingredients and moisture.

The following yields are obtained:

Non-condensable gases	10–15 per cent
Aqueous liquor	10–15 per cent
Bone tar	25–10 per cent
Bone charcoal	55–60 per cent
Total	100–100 per cent

The bone tar floating on the surface of the aqueous liquor is drawn off. It consists of fatty substances derived from the fat

which escaped extraction from the bones, also derivatives of pyridine possessing a most disagreeable and penetrating odor, and incidentally serving to distinguish it from all other tars. The characteristics of bone tar are included in Table LXXXIV.

The aqueous liquor is distilled, and the distillate caught in sulfuric acid to recover the ammonium compounds as ammonium sulfate. The residue is used as a fertilizer.

The bone tar is subjected to fractional distillation to recover the bone-tar pitch, the properties of which are also embodied in Table LXXXIV.

Bone-tar pitch is intermediate in its physical properties between asphalts and the fatty-acid pitches. On destructive distillation, it yields an aqueous distillate which reacts distinctly alkaline. It is moderately susceptible to temperature changes, and on a par with higher grades of residual asphalts and inferior grades of fatty-acid pitches in its weather-resisting properties. It is the blackest of all pitches and is therefore valued by the varnish maker to deepen the color of japans. It is soluble in pyridine bases and only partly soluble in benzol and petroleum distillates, but upon fluxing with rosin and vegetable oils it becomes soluble. Since it is produced in comparatively small quantities, it can scarcely be regarded as a commercial product.

GLYCERIN PITCH

This pitch is obtained upon the purification of glycerin by distillation with superheated steam. It is greenish brown to black in color and contains a certain amount of water-soluble constituents. It is produced in limited quantities and has a restricted use.[27]

Glycerin pitch contains polyglycerins, metallic soaps, metallic salts, etc. It is used as a lubricant in boring, for making printing inks, as a core binder,[28] etc. Upon heating with phthalic anhydride and succinic, malic, fumaric, citric, or malomalic acid, a water-resistant synthetic resin is obtained.[29]

PART IV

PYROGENOUS ASPHALTS AND WAXES

CHAPTER XX

PETROLEUM ASPHALTS

"Petroleum asphalts" are obtained from petroleums by distillation, blowing with air at elevated temperatures, and in the refining of certain distillates with sulfuric acid. These methods will be described in greater detail later.

Varieties of Petroleum. Petroleum as it occurs in different parts of the world, varies widely in composition. Certain varieties are composed of open chain hydrocarbons, others are made up exclusively of cyclic hydrocarbons, and still others occur showing every possible gradation between these two extremes. Numerous classifications have been proposed, based on its chemical composition in general, or the presence of a substantial proportion of characteristic bodies, such as the paraffin series of hydrocarbons, the naphthene series, sulfur derivatives, nitrogenous bodies, benzols, terpenes, etc.[1]

From the standpoint of the asphalt content, petroleums may be divided into three groups, viz.:

(1) *Asphaltic petroleums.* These carry a substantial amount of asphaltic bodies, with solid paraffins either absent or present only in traces.

(2) *Semi-asphaltic petroleums.* These carry a moderate amount of asphaltic bodies, but in any event generate or produce asphalt-like bodies during the distillation process. Solid paraffins may or may not be present.

(3) *Non-asphaltic petroleums.* These do not carry asphaltic bodies but may generate them during the distillation process. Solid paraffins are usually present, but not necessarily so.

TABLE XXIX—Principal Petroleum Fields of the World

Country	Name of Field	Area Included	Type of Oil	Solid Paraffins
North America				
United States	Appalachian	New York, Pennsylvania, West Virginia, Eastern Ohio, Kentucky and Tennessee	Non-asphaltic	Present
	Lima, Indiana	Michigan, Northwestern Ohio and Indiana	Non-asphaltic	Present
	Illinois	Southeastern Illinois	Semi-asphaltic	Present
	Mid-continental	Kansas; Oklahoma; northern, western and central Texas; northern Louisiana; and Southern Arkansas	Asphaltic	Absent
			Semi-asphaltic	Present
			Non-asphaltic	Present
	Gulf-coastal	Coast of Texas and Louisiana	Asphaltic	Absent
	Rocky Mountain	Colorado, Wyoming, Montana, Utah and New Mexico	Asphaltic	Absent
			Semi-asphaltic	Present
			Non-asphaltic	Present
	Northern Californian	San Joaquin Valley	Asphaltic	Absent
	Southern Californian	Los Angeles Basin and Coastal	Asphaltic	Present
Mexico	Tampico-Tuxpan	Northern Vera Cruz (including Panuco) and southern Tamaulipas	Asphaltic	Absent
	Tehuantepec	From southern Vera Cruz to eastern limit of Tabasco	Asphaltic	Variable
South America				
Venezuela	Lagunillas, La Roza and Mene Grande	Along eastern shore of Lake Maracaibo	Asphaltic	Absent
Trinidad	Trinidad	Southern portion of the island	Asphaltic	Absent
Colombia	De Barco	Along Venezuela boundary, northwest of town of Cùcuta, in the region of Barranca-Bermeja	Asphaltic	Absent
Peru	Peruvian	Along the coast from the Ecuador boundary to a point south of Payta	Asphaltic	Absent
Argentina	Comodoro-Rivadavia	Along the coast of Patagonia	Asphaltic	Absent
Europe				
Rumania	Rumanian	At Prahova, Dambovitza, Buzeu and Bacau	Semi-asphaltic	Present
			Non-asphaltic	Present
Poland	Galician	In the northern, eastern and southern foothills of Carpathian Mountains at Boryslaw Tustanowice, Solotwina and Rypne	Non-asphaltic	Variable
Russia	Groznyi	Terek Province (Northern Caucasia) at Groznyi	Non-asphaltic	Present
	Maikop	Kuban Province (Northern Caucasia) at Maikop and Bibi-Eibat	Semi-asphaltic	Present
	Caspian	North shore of Caspian Sea between the Volga and Ural Rivers in the Emba-Uralsk region	Variable	Present
Asia				
Azerbaijan	Baku	Baku Province (Transcaucasia) also the Apcheron Peninsula, including the Island of Cheleken	Non-asphaltic	Absent
		Binagadi Crude (produces petroleum asphalts which may be blown to advantage)	Asphaltic	Absent
Iraq (Mesopotamia)	Mosul	Region between the Tigris and Euphrates Rivers	Non-asphaltic	Present
Persia	Schuster	North shore of Persian Gulf (Bakhtiari region)	Non-asphaltic	Present
Siberia	Sakhalin	Sakhalin Island (Eastern Siberia)	Asphaltic	Absent
	Eastern Turkestan	Ferghana Province	Variable	Present
Japan	Kurokawa and Niitzu	Akita and Niigata Prefectures	Asphaltic	Absent
Dutch East Indies	Dutch East Indian	Islands of Borneo, Java and Sumatra	Non-asphaltic	Variable

Table XXIX contains a list of the most important occurrences of petroleum throughout the world, classified according to the asphalt content and solid paraffins present.

Different crudes yield varying amounts of asphalt on steam or vacuum distillation, dependent upon the character of the oil and the amount of asphalt content. The following figures are illustrative, and indicate in a general way the yield of asphalt by weight, having a fusing-point of 100° F. (R. and B. method):

Type of Crude Oil	A. P. I. Gravity	Yield of Asphalt
Mid-continental (Oklahoma)	42°	2 per cent
Seminole	36°	3½ per cent
Peruvian	36°	4 per cent
Mid-continental (Illinois)	32°	5½ per cent
Light Californian	26°	12 per cent
Texan	24°	14 per cent
Heavy Californian	12°	65 per cent
Mexican (Panuco)	12°	65 per cent

DEHYDRATION OF PETROLEUM

Nearly all crude petroleums carry more or less water, some being entrained mechanically, and in other cases held in a state of emulsion. Ordinarily the oil is permitted to stand long enough by itself for the water and sand to separate. Crude oils containing less than 2 per cent of water are accepted by the pipe lines and refineries. If they contain more than 2 per cent, they must first be dehydrated. Certain heavy asphaltic and waxy crudes carry as much as 70 to 90 per cent of water held in suspension by brine and colloidal mineral matter (e. g., hydrated aluminium silicates). The following methods may be used to remove the water, where it is necessary to deliver the oil to the pipe line or refinery within the prescribed limitations:

(1) *Settling.* Heating and subsidence will break up emulsions in many cases. This may be facilitated by adding chemicals, as for example 1 per cent of a mixture of sodium oleate, sodium resinate and sodium silicate; or else sulfonated oleic acid.[2]

(2) *Centrifuging.* This consists in centrifuging the oil at a temperature of 110 to 180° F. in a centrifuge operating at 17,000 r.p.m. by means of a turbine.[3]

(3) *Tube Stills.* The most satisfactory manner to break up emulsions consists in running the oil through a tube- or pipe-still under pressure and heated to 300° F., whereupon it is released in a vaporizer at atmospheric pressure. The water and oil vapors are recondensed, but will not again emulsify on account of the absence of emulsifying agents and the low viscosity of the oil.

DISTILLATION OF PETROLEUM

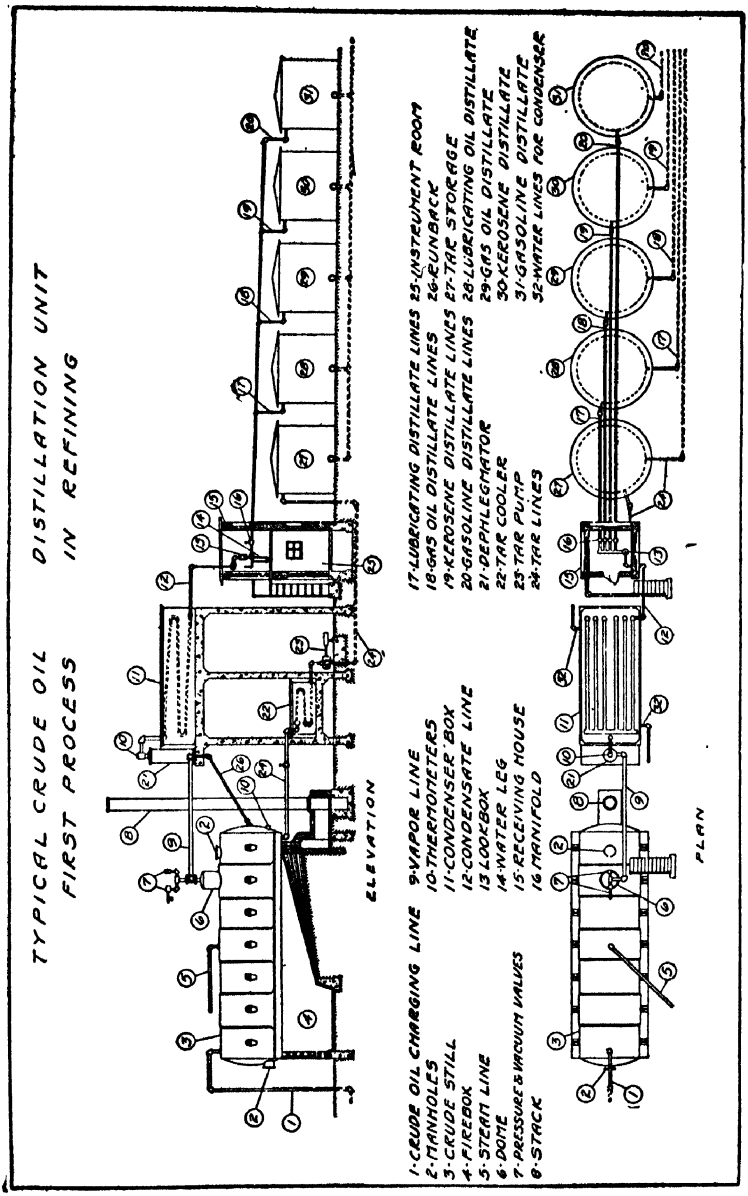
Petroleum is separated into various commercial products by means of distillation, of which the following methods are in vogue:

(1) **Batch Stills.** These are still in use in the older refineries, but are rapidly being superseded by tube stills. The efficiency in transmission of heat to the oil in the batch still is very poor, being rarely better than 30 per cent, equivalent to a consumption of 6 bbl. of fuel oil for each 100 bbl. of distillate. Approximately 0.85 bbl. distillate is produced per day, per sq. ft. of surface fired, or expressed differently, 2.5 gal. distillate will vaporize per hour, per sq. ft. vaporizing surface. Fig. 121 illustrates a typical batch-still installation. The still proper holds 1000 to 1500 bbl. of 42 gal. each. The distillation process may be carried on either with,[4] or without the use of steam. Where steam is employed, the process is known as "steam distillation," otherwise it is termed "dry distillation."

Steam Distillation. This is also known as the "fractional" distillation process and consists in introducing dry steam, termed "bottom steam" into the still, which assists in the vaporization of the volatile constituents and minimizes decomposition of the distillate and residue. Its action is based on the physical law that the boiling-point of a pair of non-miscible or slightly miscible liquids is lower than that of either pure component. The introduction of steam, therefore, serves materially to lower the boiling-point of the petroleum, and produces the maximum yield of heavy lubricating oils. It also tends to economize in fuel, and to shorten the distillation process. The steam upon being dried by passing through a trap is introduced through perforated pipes at the bottom of the still when the temperature of the contents exceeds the boiling-point of water.

In treating asphaltic petroleums by the steam-distillation process in batch stills, the charge is distilled until the residue attains the proper consistency, which is controlled by sampling the residue through petcocks set in the still, or by recording the temperature of the residue, or by observing the character of the distillate. The further the process is continued, the higher will be the fusing-point and the harder the consistency of the residue. The temperature of the residue at the termination of the process will vary between 600

and 750° F., and the time of distillation between twelve and thirty-six hours. When the distillation is completed, the residuum is run or blown into a closed cylindrical vessel constructed of steel, where



Courtesy Kansas City Testing Laboratory.

FIG. 121.—Distillation of Petroleum by Means of Batch Still.

it is allowed to cool until the temperature is reduced sufficiently to permit its being filled into barrels or drums without danger of igniting on coming in contact with air.

Where the residue is distilled to a definite consistency without further treatment, the distillation is known as "straight running," and the residue a "straight run asphalt." In certain cases a portion or "fraction" of the distillate is mixed with the residue at the close of the distillation, which is termed "cutting back," [5] the object of which is to modify the properties of the residual product or to dispose economically of a fraction which otherwise has little value commercially.

Cut-back residual oil is composed of residual oil, cut back with a petroleum distillate, which should be of a character which will volatilize within a reasonably short time after application to a surface. Relatively volatile distillates are generally used in the manufacture of cut-back asphalts. The larger the quantity of distillate added, the more fluid will be the cut-back product.[6]

In treating semi-asphaltic and non-asphaltic petroleums by the steam-distillation process in batch stills, steam is introduced when all the naphtha has distilled off, and the distillation continued until the lubricating oils have distilled over, whereupon the temperature reaches 620° F. At this point the distillation is stopped. In the case of non-asphaltic crude oil, the so-called "cylinder stock" remains in the still, and with semi-asphaltic crude, an asphaltic residue remains behind, which may either be marketed as such, or treated with air to produce a "blown asphalt."

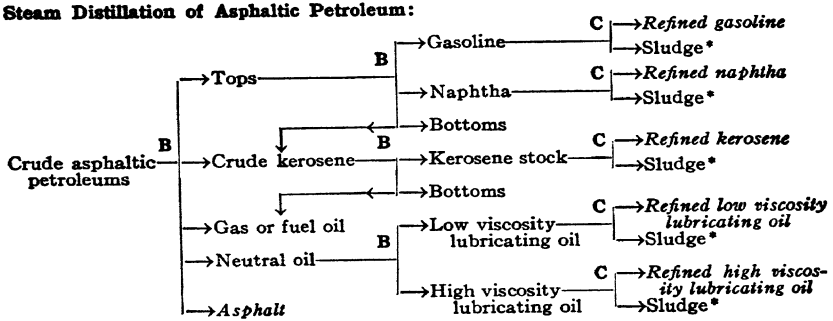
Table XXX gives an outline of the steam-distillation process as applied to asphaltic petroleum, and Table XXXI as applied to semi-asphaltic and non-asphaltic petroleums.

Dry Distillation. This is sometimes termed the "straight" or "destructive" or "cracking" distillation, by means of which a certain proportion of the higher boiling-point constituents decompose or break down, forming correspondingly larger yields of the low boiling-point constituents. The dry distillation process is accordingly used when the distiller wishes to produce a maximum amount of gasoline and illuminating oil, or in cases where the crude is unfit for manufacturing lubricating oil.

Upon treating semi-asphaltic and non-asphaltic petroleums by the dry-distillation process in batch stills, a large amount of cracking takes place when the crude kerosene fraction distils over and the temperature of the still reaches 625° F. The fires are accordingly moderated to slow down the distillation and accelerate the decomposition as much as possible. The "cracked" distillate is fractioned until the temperature in the still reaches 675 to 700° F., whereupon the distillation is brought to a close. There remains a viscous dark-colored "residuum" varying in gravity from 20 to 25° Baumé which may either be marketed under the name of "residual oil" or "flux oil" or else distilled separately in another still, known

TABLE XXX

Steam Distillation of Asphaltic Petroleum:



Legend

- **A** Dry Distillation.
- **B** Steam Distillation (or "Fractional Distillation").
- **C** Purification with Sulfuric Acid, Caustic Soda, and Water.
- **D** Filtration through Fuller's Earth.
- **E** Cooling to 20–25° F. and Filter pressing.
- **F** Sweating.
- **G** Brightening with Air jets.
- **H** Steaming.

TABLE XXXI

Steam Distillation of Non-asphaltic and Semi-asphaltic Petroleums:

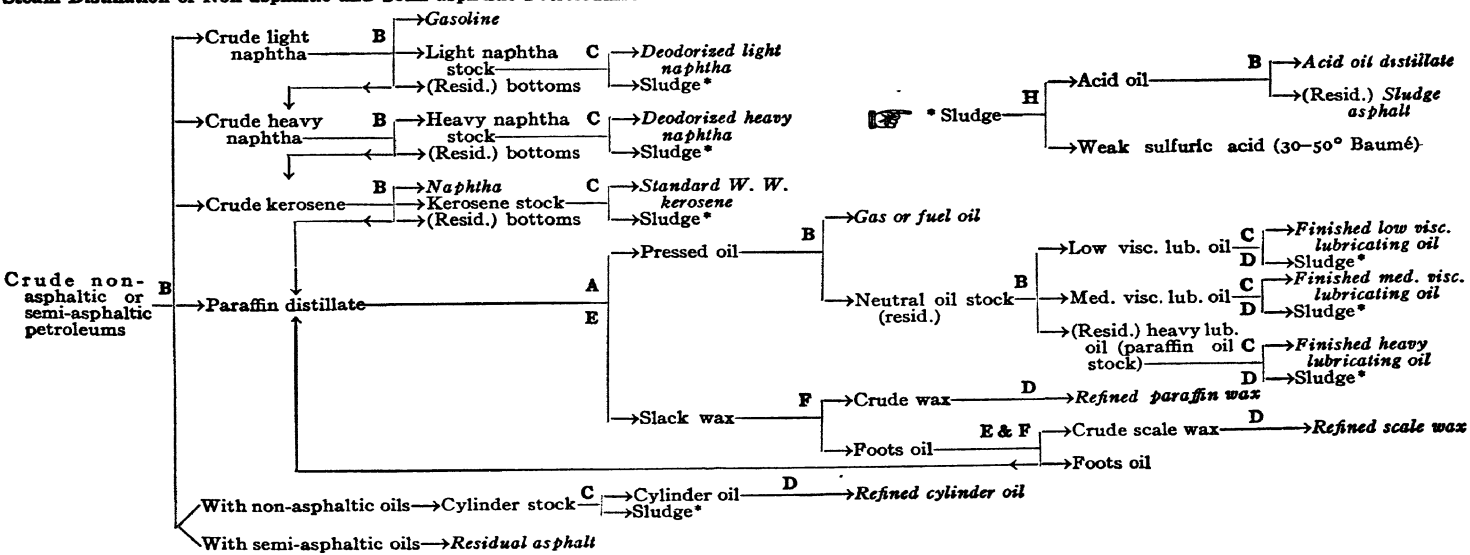
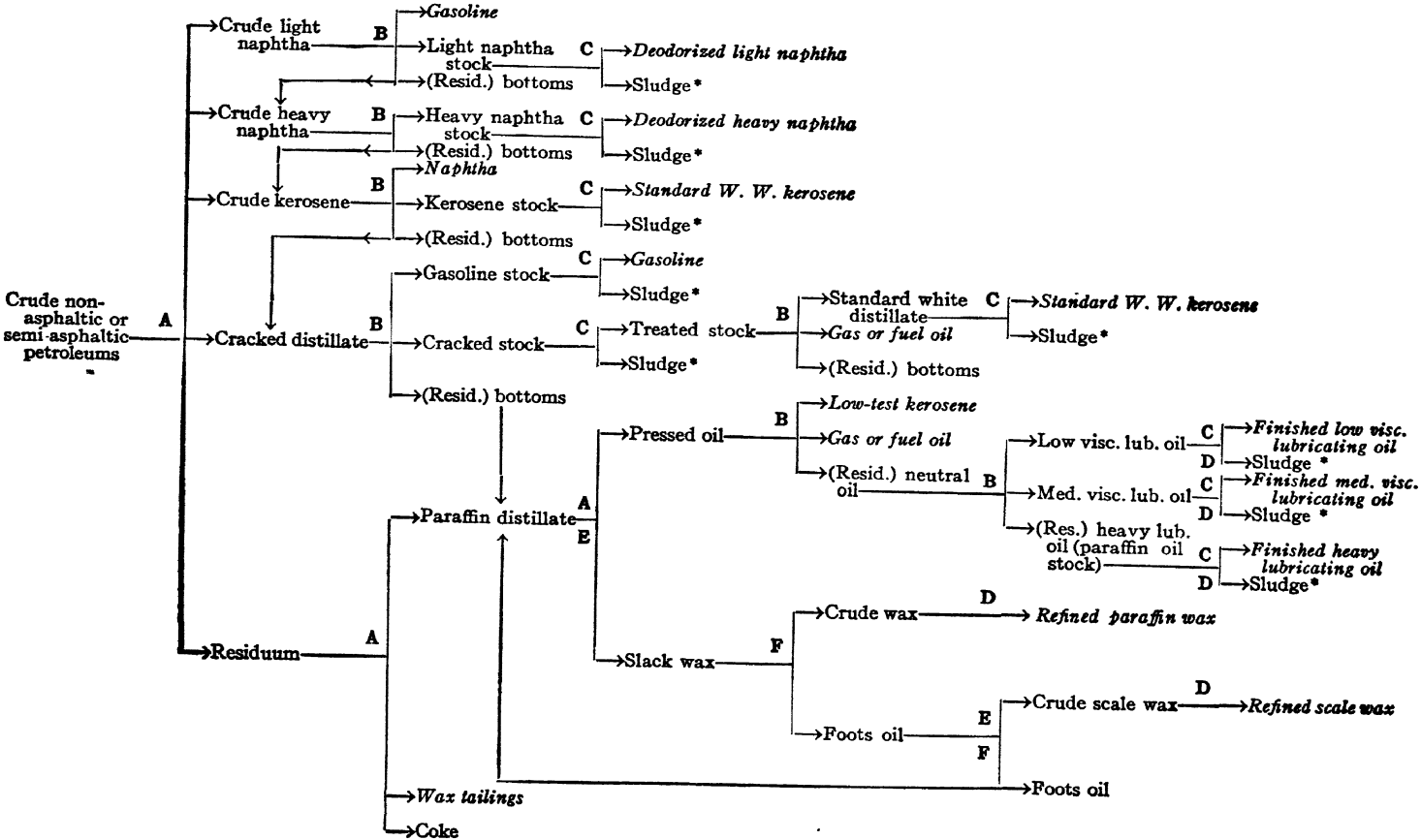


TABLE XXXII

Dry Distillation of Non-asphaltic and Semi-asphaltic Petroleums:



as a "tar still," to obtain the wax or paraffin distillate, wax-tailings and coke bottoms. The operation is carried on as rapidly as possible to avoid unnecessary cracking, and render the paraffin crystalline. The paraffin distillate comes over first, followed by the wax-tailings, until nothing but the coke remains in the still, which after cooling is removed with a pick and shovel. The method of procedure is illustrated in Table XXXII.

(2) **Continuous Stills.** Stills of this type are now encountered infrequently. They are constructed similar to batch stills, the oil flowing from one still to another, and each producing a predetermined grade of distillate. The whole circumference of a continuous still may be fired and approximately 1.42 bbl. of distillate will be obtained per day, per square foot of firing surface, similarly 2.5 gal. of distillate will evaporate per square foot of vaporizing surface. The efficiency of continuous stills is approximately 40 per cent, equivalent to a consumption of 5 bbl. of fuel oil per 100 bbl. distillate produced.

(3) **Tube or Pipe Stills.** Tube or pipe stills, also termed "vacuum flash coils" when connected with vacuum fractioning (or "flash") chambers [7] are rapidly replacing the other types of stills on account of their lower initial cost, greater through-put, and economy in operation. The oil is pumped under pressure through a series of interconnecting tubes heated in a furnace. Such stills are built in units capable of handling up to 15,000 bbl. of crude oil per day. It is possible to heat the oil as high as 900 to 950° F. because of the high rate of heat transfer through the tubes and the high velocity of the oil. This heat transfer may run as high as 200 B.t.u. per sq. ft., per hour, per degree F. difference in temperature. Their efficiency will range from 65 to 80 per cent, equivalent to a consumption of less than 3 bbl. fuel oil per 100 bbl. distillate produced.

By means of a tube still, as much as 90 per cent of the crude may be recovered as distillate and it is not infrequent that 10 per cent more gasoline will be obtained than is present in the crude oil under treatment, due to the cracking which takes place. Where more than 80 per cent of the crude is converted into distillate, there results a small amount of gas, which tends to lower the distillation temperature. The tube still has the advantage over the batch still in that the temperature may be controlled more accurately, due to the smaller volume of oil in the still, localized overheating is prevented, and the fractioning takes place within much sharper limits. Moreover, the cost of operation is from $\frac{1}{10}$ to $\frac{1}{15}$ that of batch stills, and in addition remove the possibility of recondensation and run back of the distillate. Whereas the asphaltic residue may remain at extreme temperatures for 12 to 24 hours in a cylindrical

batch still, the residue is subjected to the maximum temperature for only a fraction of a minute in a tube still, thereby minimizing any tendency towards carbonization.

Tube stills are now used successfully for all types of distillation, including the topping or skimming of crudes, the manufacture of lubricating stock, the cracking of gas oils and other distillates, and even for running the oil to coke, provided a speed of flow is attained in the tubes that the coke will not separate except in the vaporizer. They are accordingly designed to accomplish the following: dehydrate the oil and remove one or more fractions in the following order, viz.: gasoline, kerosene, gas oil and/or wax-distillate (in the case of semi-asphaltic and non-asphaltic petroleums). The residue consists either of fuel oil or residual oil, depending upon the extent to which the distillation has been carried.

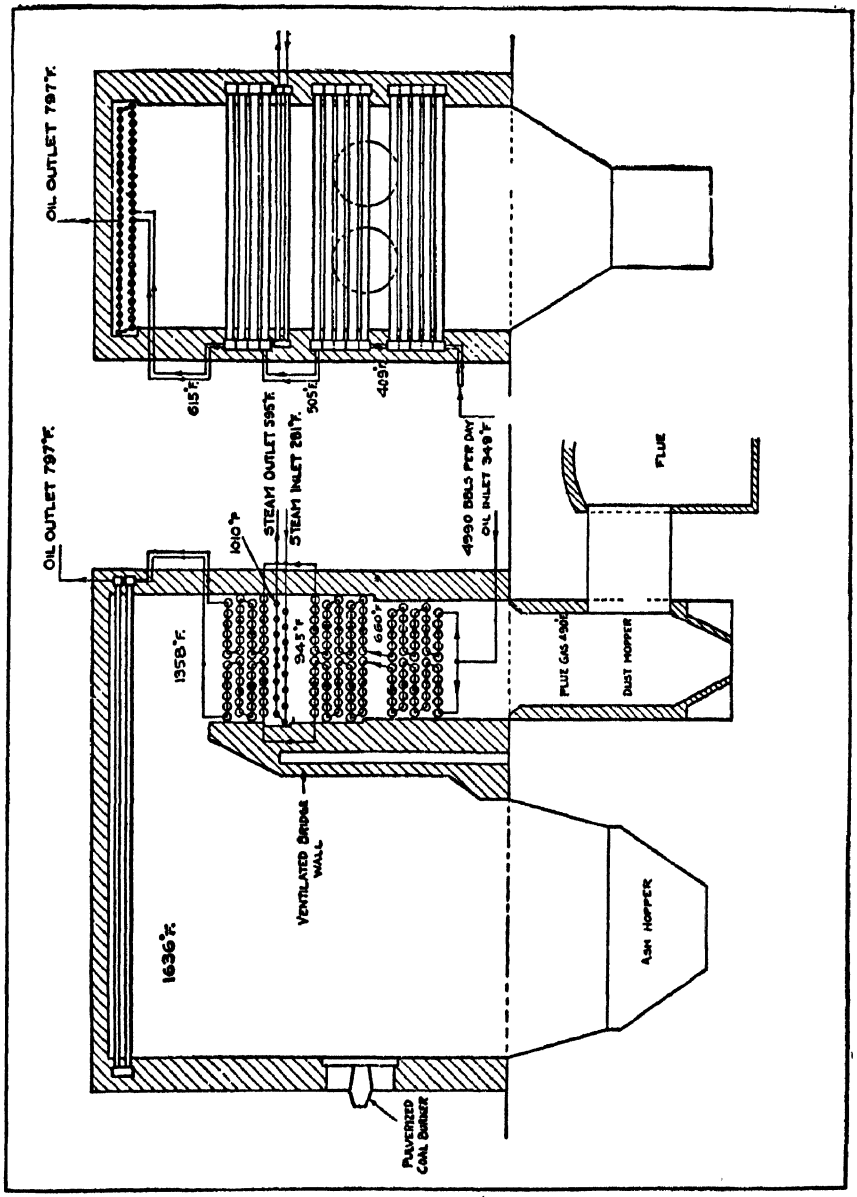
The construction details of a tube-heater is shown in Fig. 122.

The successful operation of the tube still is closely connected with the improved fractioning equipment developed during the past five to ten years. It is now possible to produce fractions of the proper distillation range in one operation. The so-called bubble-cap tray-tower has given phenomenal results. It consists in an arrangement for the repeated absorption and redistillation of the distillate, thus separating the heavier hydrocarbons, which are carried over by the associated lighter hydrocarbons. Such a tower is illustrated in Fig. 123. The tower is insulated to keep it at an unvarying temperature and means are provided to keep a definite relation between the up-flowing vapors and the down-coming liquid which flows from tray to tray in the tower. It has been found that very volatile distillates can only be fractioned at high pressures—usually 250 lb. per sq. in.; and moreover difficultly volatile distillates can be fractioned most closely in towers operating under a vacuum of 29, or in some cases above 29¾ in.[8]

The general arrangement of a two-stage combination atmospheric and vacuum distillation unit for treating 10,000 barrels of crude in 24 hours is illustrated in Fig. 124.[9] This procedure involves the recovery of the lighter distillates, including naphtha, kerosene and a portion of the gas oil by distillation at atmospheric pressure, and thereupon transferring the residue to a vacuum distillation unit in which the balance of the gas oil and several lubricating-stock fractions are recovered as distillates and asphalt as a final residuum.

The general operation of this unit is as follows: crude is taken from storage and pumped through the atmospheric vapor heat ex-

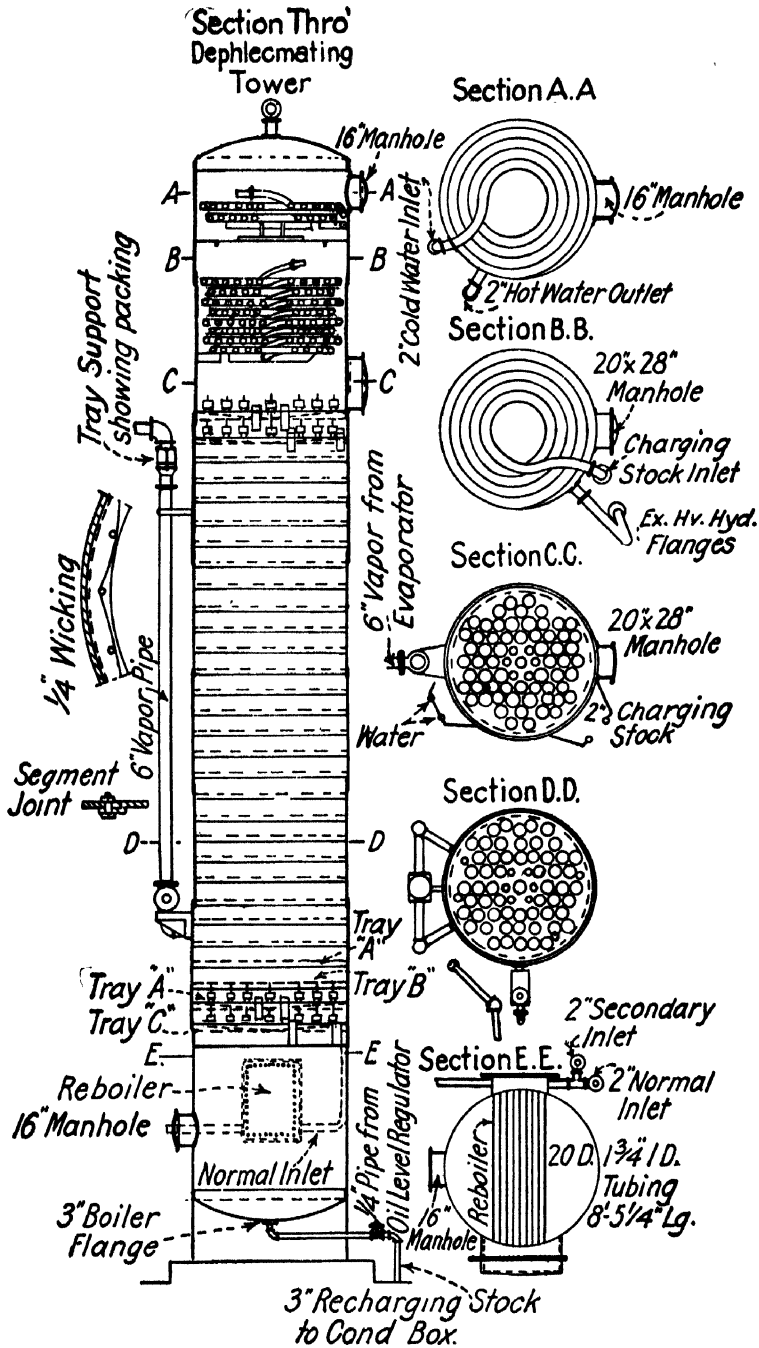
changer and thence to a settling tank for removal of solids and water. The crude then flows through the vacuum-stage exchangers, acquiring a final preheat temperature of approximately 350° F.



Courtesy Kansas City Testing Laboratory.

FIG. 122.—Sectional View of a Tube Heater.

The preheated crude is transferred to the atmospheric stage tube still and heated to a temperature sufficient to effect the desired vaporization in the atmospheric fractioning tower.



Courtesy Kansas City Testing Laboratory.

FIG. 123.—Bubble-cap Tray-tower for Fractioning Petroleum.

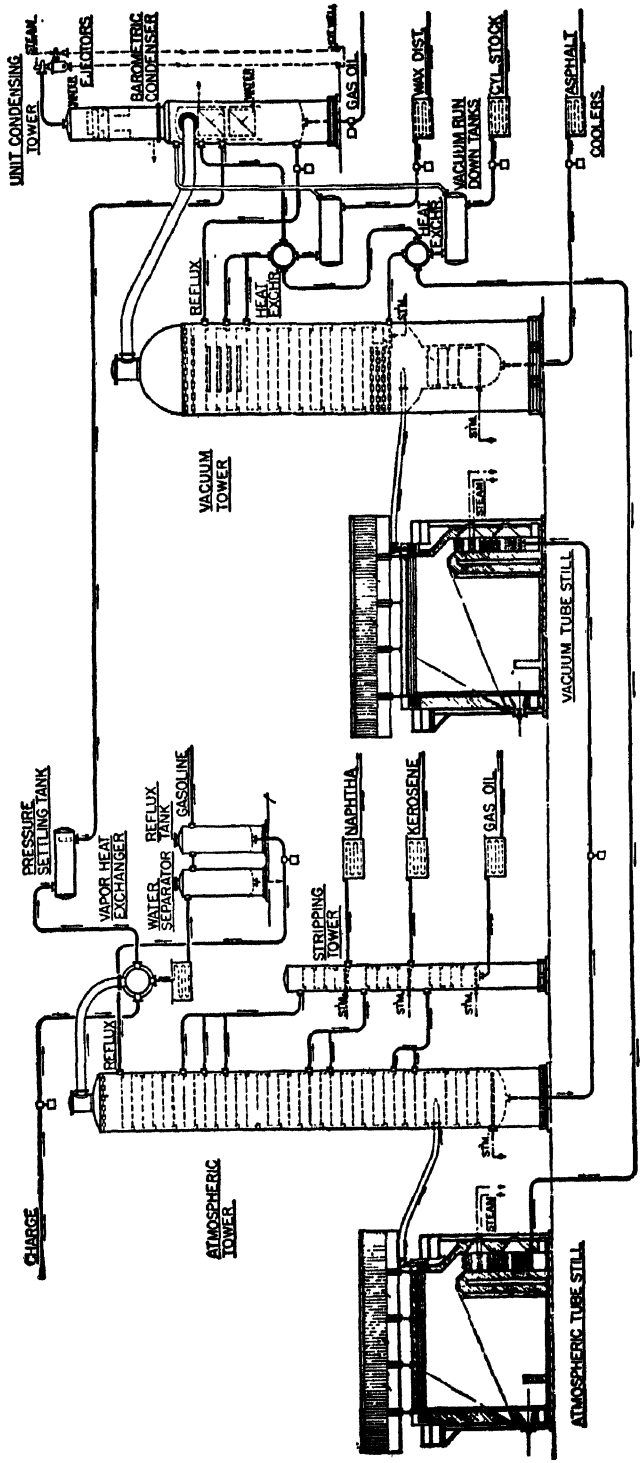


FIG. 124.—Two-stage Atmospheric and Vacuum Distillation Unit.

In the atmospheric tower four distillate streams are recovered: gasoline as the top vapor stream, and naphtha, kerosene, and gas oil as liquid side streams.

The side streams are removed from respective plates in the main tower to external stripping sections for steam stripping to remove the lighter ends. The stripping steam and vapors are returned to the main tower at points above the respective stream take-offs.

The atmospheric stage bottoms are subjected to steam stripping on plates below the tower inlet. The reduced crude is pumped hot from the base of the atmospheric tower directly to the vacuum-stage tube still for further heating.

The vacuum tube still discharges into the vacuum fractionating tower, which is maintained at a low absolute pressure. Three distillate streams are recovered from the vacuum stage: heavy gas oil as the top vapor stream and paraffin distillate and cylinder stock as liquid side streams. Asphalt bottoms are recovered as a residue from the base of the tower. Steam is introduced into the base of the tower for stripping the asphalt and for partial-pressure effect.

Vacuum is maintained on the system by a barometric condenser backed by steam jet ejectors. The overhead vapor stream, consisting of gas oil and process steam, passes through vapor heat exchangers and water-cooled surface condensers. Because of the temperature and pressure conditions maintained at the condenser outlet, the process steam remains in the vapor state and flows to the barometric condenser, while practically complete condensation of the gas oil is obtained. The desirability of obtaining sharp phase separation at the vacuum condenser outlet with a low partial pressure of gas oil vapor in the uncondensed steam, dictates the quantity of gas oil to be removed in the atmospheric stage; i.e., the initial boiling point of the vacuum-stage gas oil must be sufficiently high to insure substantially complete condensation.

Process conditions and sizes of the major items of equipment for this installation are shown in Table XXXIII.

Two-stage crude distillation units with individual unit capacities up to 50,000 barrels per day are now under construction. Based on 330 stream days per year, 50,000 barrels per day correspond to 16,500,000 barrels of crude oil per year. These figures indicate

the enormous capacity which can be built into single process units of this type.

The two-stage units effect a separation between asphalt and lubricating stocks by distillation. Because of overlapping in boiling point range between the heaviest lubricating fraction and the lower boiling asphaltic material, a sharp separation is difficult. Hence it

TABLE XXXIII

TWO-STAGE DISTILLATION UNIT

Crude charge, bbl./24 hr.	10,000
Vacuum-stage charge, bbl./24 hr.	5,000
Final asphalt residuum, bbl./24 hr.	837
Temp., ° F.:	
Atmospheric tower inlet	599
Atmospheric tower top	224
Atmospheric naphtha draw-off	328
Atmospheric kerosene draw-off	420
Atmospheric gas oil draw-off	537
Atmospheric bottoms draw-off	530
Vacuum tower inlet	772
Vacuum tower top	364
Vacuum tower light lube draw-off (wax dist.)	504
Vacuum tower heavy lube draw-off (cylinder stock)	690
Vacuum tower bottoms draw-off	709
Absolute pressure at top of vacuum tower, mm.	60
Atmospheric tower:	
Diam., ft.	11
No. plates (30 above inlet, 4 below)	34
Vacuum tower:	
Diam., ft.	17
No. plates (16 above inlet, 4 below)	20
Process steam, lb./hr.:	
Atmospheric tower and strippers	3,500
Vacuum tower	4,500
Vapor velocity:	
Atmospheric tower:	
Ft./sec.	2.21
Lb./sq. ft./hr.	1,360
Vacuum tower:	
Ft./sec.	7.05
Lb./sq. ft./hr.	600

has been found advisable in some instances to remove a clean, heavy, lubricating stock several trays above the flash zone and a small quantity of material containing the lighter asphaltic materials as a total draw-off from the tray immediately above the tower inlet. This stream can be treated separately from the clean bulk heavy lubricating stock, or if the heavy lubricating available in the crude exceeds requirements, it can be routed to cracking.

Several lubricating-stock rerun operations are commonly practiced. The modern equipment in general use consists of atmospheric or vacuum pipe stills similar in design to those used for crude units. Major operations are as follows:

Rerunning of Pressed Wax Distillate. This operation consists in the redistillation of stock which has been dewaxed by filter pressing. The rerun operation eliminates non-viscous gas oil, and the balance of the dewaxed oil is fractioned into two or more neutral oils for use in blending for automotive lubricant or in the manufacture of light machine oils. Atmospheric distillation is customarily employed with tube still outlet temperatures of 700° to 730° F.

Cylinder Stock Solution Rerunning. The cylinder stock-naphtha mixture from centrifuge dewaxing is rerun in atmospheric tube stills for removal of the naphtha diluent. Tube-still outlet temperatures of 475° to 550° F. are required.

Rerun of Treated and Dewaxed Long Residuum for Motor Oil Grades. A vacuum operation is dictated by the fact that lubricating fractions of high boiling point are taken overhead.

CRACKING PROCESSES

These have been developed for the purpose of increasing the yield of gasoline, which is accomplished by heating the crude oil to a high temperature under pressure.

(1) Liquid Phase Cracking. This involves heating and cracking at high pressures without permitting distillation to take place during the reaction stage. The heat is accordingly applied to the liquid under a sufficiently high pressure to maintain it as a liquid and prevent the distillation and consequent drop in temperature in the reaction chamber. These processes usually operate continuously, with separate heating and reaction chambers.

(*a*) *Tube-and-Tank Process.* This is illustrated diagrammatically in Fig. 125, and consists of a tube heater *A* in which the oil enters under a pressure of 400 lb. per sq. in. At the exit the pressure is 350 lb. and the temperature 850° F. The oil then enters an insulated vertical reaction chamber *B*, which is kept filled with liquid under 350 lb. pressure, and at its exit the temperature has fallen to 785° F. Before it enters the bubble tower *C*, the pressure is reduced to 50 lb. by the release valve *D*, and its temperature as it enters the tower is 700° F. The bubble tower separates the gas oil (i.e., cycle stock) and the pressure tar. The cycle stock is run through the process again, mixed with a certain proportion of fresh

crude oil. The gasoline is then condensed and separated from the fixed gas which is consumed as part fuel in the tube heater.

(*b*) *Cross Process.* This is illustrated in Fig. 126 and consists of two tube heaters *A* and *B* using common vaporizing and fractioning towers *D* and *E*. The first heater operates on crude oil, or fuel oil, or in fact any petroleum product and heats the charging stock to 850° F. under 350 lb. pressure. The second heater *B* operates on the recycling stock, which it heats to 900° F. under 600–700 lb. pressure. From here the oil is forced into an insulated reaction chamber *C*, where it is maintained in liquid condition for fifteen minutes at a temperature of 600–700° F., during which all the carbon formed in the cracking operation is deposited as coke, which must be cleaned out from time to time. At the exit of *C* there is

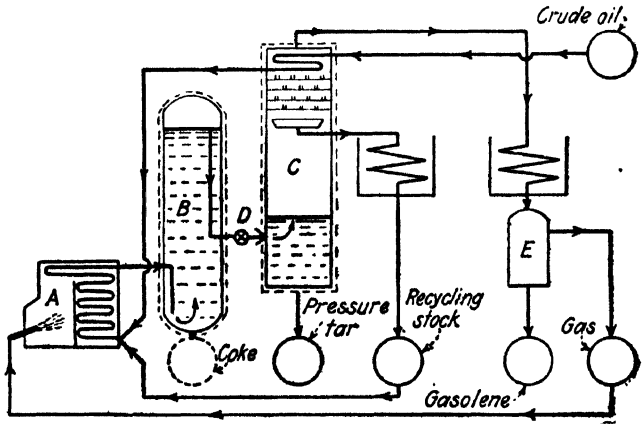


FIG. 125.—Tube-and-Tank Cracking Process.

a reducing-valve which discharges the product into a vaporizer *D* maintained at practically atmospheric pressure. As the cracked oil enters the vaporizer, it blends continuously with the stream from the tube still *A*. The fractioning tower *E* separates the recycling stock (gas oil), and the gas-separator *F* separates the gasoline from the gas. Tube still *A* acts as a skimming plant which separates the gasoline normally present in the crude oil, plus gas-oil, plus pressure-tar. The final operation results in the production of gasoline, recycling stock (gas oil), pressure-tar, gas (which is burned underneath the tube stills), and carbon (which must be periodically removed from the reaction chamber). The pressure-tar may be marketed as fuel oil, since it is very fluid, and practically free from carbon (less than ½ per cent). A plant of this type will operate successfully on all types of crude oil, emulsified crudes, as well as any fraction distilled therefrom.

(*c*) *Holmes-Manley Process.* This is very similar to the Tube-and-Tank Process, the only material difference being that four or more vertical reaction chambers are mounted side by side in a common gas-fired furnace, and in addition each reaction chamber is provided with a motor-driven shaft having scrapers to assist in removing the coke. The oil enters the tube heater under 350 lb. pressure and leaves at 300 lb. and 800° F. A reducing-valve is

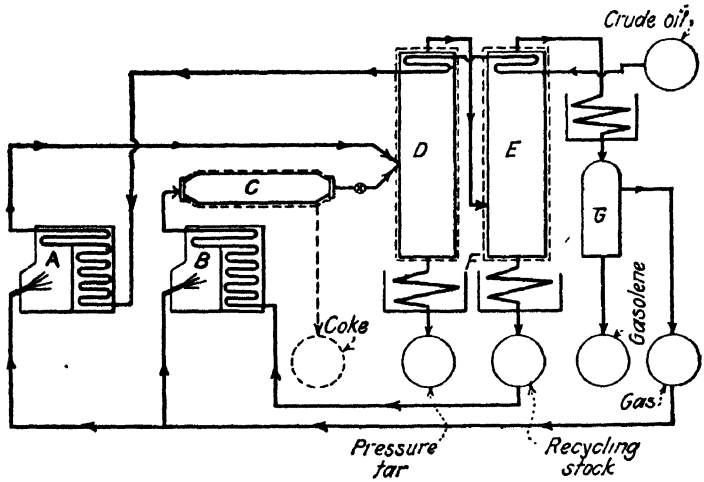


FIG. 126.—Cross Cracking Process.

located between the condenser and the gas-separator in which a pressure of 80 lb. is maintained.

(2) Mixed Phase Cracking. This involves pressure distillation with simultaneous heating, cracking and distillation. The efficiency is stated to be less than in the liquid phase cracking, because of the added heat consumed in the repeated vaporization and condensation of the uncracked distillates.

(*a*) *Burton Process.* This is a batch operation and takes place in stills with refluxing. The modern high-pressure Burton still [10] consists of a pair of heat-insulated cylinders *A* (Fig. 127), attached to a tube heater *B*, mounted in a furnace below. This is connected with a bubble-tower *C*, which separates the gas and gasoline from the heavier distillate which is allowed to recycle into the still. Condenser *D* separates the gasoline, which in turn is separated from the gas in the gas-separator *E*. The entire system up to the release-valve *F* is maintained under a pressure of 925 lb. The still *AB* is heated to 750° F. and the bubble tower *C* to 550° F. The carbon

assumes a granular condition and is cleaned out after each run of forty-eight hours, whereupon the still is recharged. Upon running gas-oil there are produced 80 per cent gasoline, 6 per cent gas, 0.4 per cent coke and 13.6 per cent pressure-tar. The pressure-tar

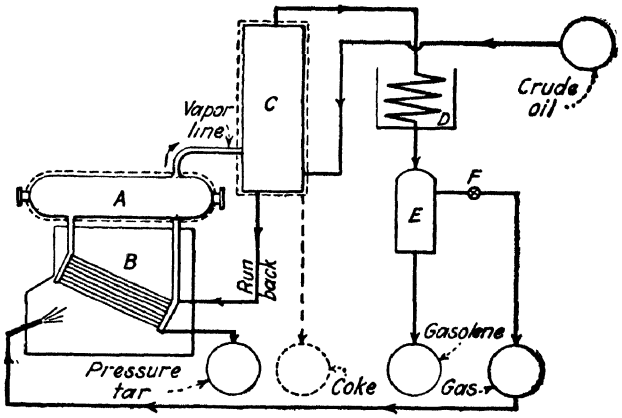


FIG. 127.—Burton Cracking Process.

carries but a small amount of carbonaceous matter and when subjected to steam-distillation in a batch still produces a hard asphalt of high ductility, almost completely soluble in carbon disulfide, and

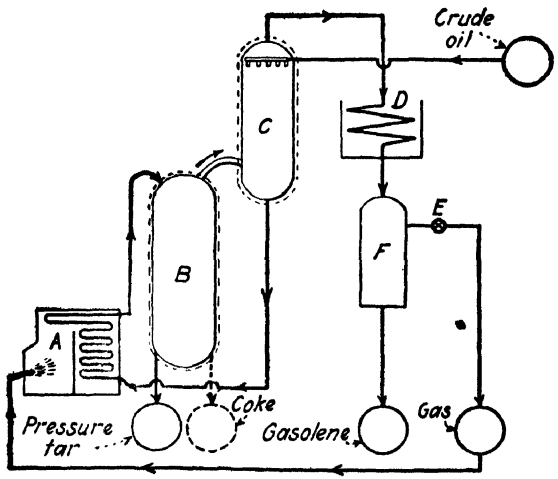


FIG. 128.—Dubb's Cracking Process.

which is quite susceptible to temperature changes.[11] This has been marketed in the United States under the name "Stanolite," which, according to the experience of the author, is lacking in weather-resisting properties.

(*b*) *Dubb's Process.* This process operates continuously at a high temperature. The lay-out is illustrated in Fig. 128 and consists of a tube still *A* heated partly with the fixed gas produced in the cracking operation and partly by means of fuel oil, which raises it to 850–900° F. under a pressure of 250 lb. The product thereupon enters the reaction chamber *B* in which it is vaporized, accompanied by a drop in temperature and deposition of coke. The vapors next pass into a dephlegmator *C* where they encounter a stream of the raw charging stock and upon mixing pass downward and back to the tube still *A*. The uncondensed vapors pass out at the top of the dephlegmator and then into a coil-condenser *D*. The valve *E* releases the pressure and the separator *F* removes the gas from the gasoline. Where it is desired to produce pressure-tar (in this case residual oil), it is drawn off the bottom of the reaction chamber *B*, otherwise the valve is kept shut and a slightly higher temperature is carried on the transfer liquid. The hot residue may be quenched by running into water and separating.[12] The foregoing process results in the production of cracked vapor and coke, instead of cracked vapors, coke and residual oil. The following are reported to have been obtained on one cycle:

From Mid-continental gas oil	44.3 per cent gasoline
From Mid-continental fuel oil	40.9 per cent gasoline
From Panuco 10° Bé. residuum	21.1 per cent gasoline
From Venezuela 13° Bé. residuum	26.9 per cent gasoline

The various cracking processes ranked as follows in the United States in relation to the respective through-puts: Burton, Cross, Holmes-Manly, Tube-and-Tank and Dubbs. The following types of stock may be used in the cracking operations: crude oils of any type, emulsified crudes of any type, topped crudes, fuel oil, gas oil, and mixtures of the foregoing with recycling stock. The following in general represent the products obtained: fixed gases (used as fuel), gasoline, pressure-tar and in special cases an intermediate cut known as recycling stock (which is retreated and still further cracked), also coke. After removal of the soluble oils, the coke tests as follows: volatile matter 13 per cent, fixed carbon 80 to 86 per cent, ash 1 to 7 per cent, and sulfur 1 to 2 per cent.

The pressure-tar may be utilized in the following ways, viz.: marketed as fuel oil (provided it is sufficiently liquid and free from carbonaceous matter); or it may be marketed as soft asphalt flux (provided it is more viscous than fuel oil); or it may be mixed in suitable proportions with residual asphalts derived from the regular distillation process;[13] or it may be steam-distilled in a batch still to produce residual asphalt of various grades; or distilled under vacuum to the desired grade;[14] or it may be blown with air to pro-

duce blown asphalts of the desired grade, either alone,[15] or in admixture with other products, such as an "aromatic extract of a petroleum oil," [16] or an untreated residual oil in combination with an asphalt of naphthene base,[17] or the oily layer separated from sludge asphalt.[18] Pressure-tars may be hydrogenated under pressure at 345–400° C. in the presence of a catalyst.[19]

Various tars from coal (e. g., low-temperature coal tar, water-gas tar, coke-oven tar, etc.), shale, lignite, peat, or wood, may likewise be treated by the cracking process.[20] The following figures will indicate the yields when such tars are submitted to the cracking process:

	Spec. Grav. (A. P. I.°)	Gas Cu. ft. per bbl.	Motor Fuel Per Cent	Fuel or Diesel Oil Per Cent	Coke. Lbs. per barrel
Low-temperature coal-tar.	8.9	570	28.8	10.3	...
Intermediate temperature coal-tar.....	6.0	500	14.5	17.7	241
High-temperature coal-tar.	7.0	706	12.1	22.6	158
Lignite tar.....	13.9	767	38.3	....	241
Brown-coal tar.....	9.2	835	28.6	....	96

The following constitute the various products pertaining to, or derived from petroleum: [21]

Liquid Products

Petroleum Naphtha. A generic term applied to refined, partly refined or unrefined petroleum products and liquid products of natural gas, not less than 10 per cent of which distils below 347° F. (175° C.) and not less than 95 per cent of which distils below 464° F. (240° C.), when subjected to distillation in accordance with the standard method of test (A.S.T.M. Designation: D 86).

NOTE.—The "naphthas" used for specific purposes such as cleaning, manufacture of rubber, manufacture of paints and varnishes, etc., are made to conform to specifications which may require products of considerably greater volatility than that set by the limits of this generic definition.

Gasoline. A refined petroleum naphtha which by its composition is suitable for use as a carburant in internal combustion engines.

Petroleum Spirits (White Spirits). A refined petroleum distillate with a minimum flash point of 70° F. (21° C.) determined by the Tag Closed Tester (A.S.T.M. Designation: D 56), or by the Abel Tester, with the volatility and other properties making

it suitable as a thinner and solvent in paints, varnishes and similar products.

NOTE.—The term "turpentine substitute" as applied to petroleum spirits is to be condemned as false and misleading. The term "mineral spirits" which is frequently used in the paint and varnish industry is a misnomer as it includes within its scope not only petroleum products, but other hydrocarbon mixtures, such as coal-tar distillates. In Great Britain the term "petroleum spirits" is applied to a very light hydrocarbon mixture having a flash point below 32° F. (0° C.).

Engine Distillate. A refined or unrefined petroleum distillate similar to naphtha, but often of higher distillation range.

Tops. The unrefined distillate obtained in topping a crude petroleum.

Kerosene. A refined petroleum distillate having a flash point not below 73° F. (23° C.), as determined by the Abel Tester (which is approximately equivalent to 73° F. (23° C.) as determined by the Tag Closed Tester; A.S.T.M. Designation: D 56), and suitable for use as an illuminant when burned in a wick lamp.

NOTE.—In the United States of America local ordinances or insurance regulations require flash points higher than 73° F. (23° C.), Tag Closed Tester.

Gas Oil. A liquid petroleum distillate having a viscosity intermediate between that of kerosene and lubricating oil.

NOTE.—It should be understood that oils, other than gas oil as defined above, may be and are used in the manufacture of gas.

Fuel Oil. Any liquid or liquefiable petroleum product burned for the generation of heat in a furnace or firebox, or for the generation of power in an engine, exclusive of oils with a flash point below 100° F., Tag Closed Tester, and oils burned in cotton or wool-wick burners. Fuel oils in common use fall into one of four classes: (1) residual fuel oils, which are topped crude petroleums or viscous residuums obtained in refinery operations; (2) distillate fuel oils, which are distillates derived directly or indirectly from crude petroleum; (3) crude petroleums and weathered crude petroleums of relatively low commercial value; (4) blended fuels, which are mixtures of two or more of the three preceding classes.

Lubricating Oils. A heavy distillate suitable for use in lubrication.

Semi-Solid to Solid Distillates

(*a*) *Petrolatum.* This term is applied to the product obtained by diluting either crude paraffinaceous petroleum, or the residue known as "cylinder stock" (obtained when paraffinaceous petroleum has been steam-distilled to remove the lighter distillates and gas-oil) with naphtha. The mixture is then chilled or allowed to "cold

set" until an amorphous residue settles out, which is separated either by sedimentation or by centrifuging. The naphtha carried by the petrolatum is recovered by steam distillation. Petrolatum is also known under the names vaseline, petroleum grease, petroleum jelly, and liquid paraffin.

(*b*) *Paraffin Wax.* This is derived from paraffin-bearing petroleums, and is separated from the lubricating oil and paraffin distillates by crystallization at low temperatures and filter-pressing. It is classified by its color and melting-point. The terms "paraffin scale" and "scale wax" are generally applied to the low melting-point variety, and "refined paraffin wax" to the harder variety. Its melting-point varies from 100–135° F.

(*c*) *Wax Tailings.* This represents the fraction obtained in the dry distillation of petroleums, and recovered immediately prior to coking. It is peculiar in its properties and generally free from paraffin wax.

Semi-Solid to Solid Residues

(*a*) *Residual Oil.* This term is applied to the liquid to semi-solid residues obtained from: (1) the destructive distillation of non-asphaltic petroleum; (2) the distillation of semi-asphaltic and asphaltic petroleums; (3) the distillation of pressure-tar; (4) the fluxing of harder residual asphalts with heavy distillates (known as "cut-back asphalt"). The following terms are used synonymously with residual oil, viz.: asphaltum oil, liquid asphalt, flux, flux oil, fluxing oil, roofing flux, road oil, dust-laying oil, black oil and petroleum tailings; and abroad as mazout, mazut and masut (Tartar), ostatki or astatki (Russian), also pacura (Rumanian).

(*b*) *Residual Asphalt.* This term is applied to the semi-solid to solid residues obtained from the distillation of semi-asphaltic and asphaltic petroleums and pressure-tars. The following terms are used synonymously with residual asphalt, viz.: petroleum asphalt, petroleum pitch, petroleum residue, road binder, carpeting medium, and seal-coating material.

(*c*) *Blown Asphalt.* A term used to designate the product obtained by blowing air through residual oil at elevated temperatures. Also known under the names oxidized asphalt, oxygenized asphalt, oxygenated asphalt and condensed asphalt.

(*d*) *Sulfurized Asphalt.* This term is applied to the product obtained by heating residual oil or residual asphalt with sulfur at high temperatures. Also known under the names Dubbs asphalt, Pittsburgh flux and Ventura flux.

(*e*) *Sludge Asphalt.* This term is applied to the asphaltic product separated from the acid sludge produced in refining petro-

leum distillates with sulfuric acid. It is also known under the names acid-asphalt, acid-sludge asphalt, etc.

RESIDUAL OILS

Residual oils are distinguished from fuel oils by having a higher specific gravity, a higher fusing-point and generally a smaller amount of volatile matter and higher flash-point (except in the case of cut-back residual oils). The two classes of products merge together, so that there is no sharp line of demarcation between them.

The characteristics of residual oils depend upon three factors, viz.:

(1) The type and nature of the petroleum from which they are produced.

(2) The particular form of apparatus in which the distillation is carried out.

(3) The extent to which the distillation has been conducted, and whether or not the product has been cut back.

The following will indicate in a general way the effect of these factors upon the nature of the residual oil.

Fusing-point. All types of residual oil fall within the range of 32 to 110° F., R. and B. method (Test 15*b*); or 20 to 100° F., K. and S. method (Test 15*a*).

Specific Gravity. The type of petroleum and the nature of the distillation process affects the specific gravity at 77° F. (Test 7) as follows:

	When Straight-distilled	When Distilled from Pressure-tar
From non-asphaltic petroleum	0.95–0.95	1.00–1.03
From semi-asphaltic petroleum	0.90–1.00	1.02–1.05
From asphaltic petroleum	0.95–1.02	1.03–1.07

Solid Paraffins. The percentage of solid paraffin (Test 33) is an indication of the types of petroleum from which the residual oil has been produced, regardless of the particular form of apparatus in which the distillation has been carried out. Thus, residual oils derived from non-asphaltic petroleums will contain from 4.0 to 15.0 per cent solid paraffin, those derived from semi-asphaltic petroleums from a trace to 5.0 per cent, and those derived from asphaltic petroleums from 0.0 to 0.25 per cent.

Fixed Carbon, Sulfur, Saturated Hydrocarbons and Proportion Soluble in 88° Petroleum Naphtha. The following means may be used to differentiate between residual oils derived from various sources: [22]

Residual Oils from:	Fixed Carbon (Test 19)	Sulfur (Test 28)	Saturated Hydrocarbons (Test 34a)	Soluble 88° Naphtha (Test 23)
	Per Cent	Per Cent	Per Cent	Per Cent
U. S. non-asphaltic petroleum	<5	<1.5	>75	>90
U. S. semi-asphaltic petroleum	<5	<1.5	50–75	>90
U. S. asphaltic petroleum.....	5–7	1.5–2.5	30–40	80–95
Trinidad, Venezuela and Colombian petroleums.....	7–10	2.5–3.5	30–40	80–95
Mexican petroleum.....	>10	3.5–5.0	30–40	80–95

The foregoing figures apply to the residual oils produced on straight distillation as well as from the corresponding pressure-tars.

Flash-point and Volatile Matter. These two criteria are closely related and interdependent. The lower the flash-point, the greater will be the percentage of volatile matter, and vice versa. Both fluctuate within wide limits in the case of residual oils. Excepting for cut-back products, the flash-point of residual oils is generally above 300° F. Cut-back products may run as low as 200° F., which is likewise the case with fuel-oils and undistilled pressure-tars. Similarly the volatile matter at 500° F. in five hours will generally run below 15 per cent or at most 20 per cent, except in the case of cut-back products, which will run in excess of 20 per cent. Where residual oils are used to flux harder asphalts and asphaltites, or where they are converted into blown asphalts, it is desirable that the flash-point shall be as high as possible (preferably above 450° F.) and the volatile matter as low as possible (preferably below 3 or 4 per cent at 500° F. in five hours). On the other hand, if the residual oil is to be used for dust-laying purposes the presence of volatile constituents is not detrimental, but on the contrary may be desirable, and hence cut-back products will answer satisfactorily. Residual oils produced from Colombian petroleum (asphaltic) show the highest flash-point and the smallest amount of volatile matter. In fact, Colombian petroleum is so difficultly volatile that it must be distilled under vacuum after the lower boiling fractions have been removed by topping in a shell or pipe still.[23] Residual oils produced from Venezuela and Trinidad (asphaltic) petroleums, also U. S. Mid-continental (semi-asphaltic) and U. S. Gulf-

coast (asphaltic) show a somewhat lower flash-point and a correspondingly greater amount of volatile matter. Residual oils obtained from Mexican (asphaltic) and U. S. Californian (asphaltic) petroleums show a still lower flash-point and greater quantity of volatile matter than the preceding, basing the comparisons on products of similar fusing-points and hardness (viscosity).

Residual oils from non-asphaltic petroleums are not produced today in appreciable quantities, as this grade of petroleum is usually distilled destructively to recover the lubricating oils and paraffin wax. Moreover, such residual oils have certain inherent disadvantages, including the fact that they are poor fluxes, they cannot be blown successfully, and they contain a quantity of greasy and waxy constituents which contaminate any other substances with which they may be combined. Residual oils derived from asphaltic petroleums constitute the best fluxes and produce the most desirable grades of blown asphalts. Those produced from pressure-tars will show evidence of free carbon under the microscope and sometimes also to the naked eye, and it is contended that unless used in admixture with other products, they are undesirable for producing blown asphalts, since they are likely to result in a non-homogeneous product. It is contended, however, that pressure-tars may be blown with a mixture of air and steam at temperatures of 450–550° F., above which temperature carbon will be generated.[24] Any free carbon may be removed by filtering the tar;[25] by mixing the pressure-tar with fuel oil, heating to 340° F. and either filtering or centrifuging the mixture;[26] by treatment with sulfuric acid;[27] by the addition of CaO during the cracking process, which results in an agglomeration of the carbon and facilitates the filtration.[28]

Another residual oil, which may suitably be used to flux harder asphalts, consists in the semi-liquid residue produced in the refining of lubricating oils with selective solvents.[29]

Residual oils, with the exception of cut-back products, comply with the following characteristics:

(Test 1)	Color in mass	Brownish black to black
(Test 2*a*)	Homogeneity to the eye	Uniform to gritty
(Test 2*b*)	Homogeneity under microscope	Uniform to lumpy
(Test 7)	Specific gravity at 77° F.	0.85–1.07
(Test 8*a*)	Specific Engler viscosity at 140° F.	10– 50
(Test 9*b*)	Penetration at 77° F.	100–350
(Test 9*c*)	Consistency at 77° F.	0– 7
(Test 15*a*)	Fusing-point K. and S. method	20–100° F.
(Test 15*b*)	Fusing-point R. and B. method	32–110° F.
(Test 16)	Volatile at 325° F. in 5 hours	½–12 per cent
	Volatile at 500° F. in 5 hours	1–20 per cent

(Test 16*a*)	Residue of 100 penetration at 77° F. obtained on evaporation	40–80 per cent
	Ductility of residue at 77° F. (Test 10*a*)	2–100 + cm.
(Test 17*a*)	Flash-point	300–550° F.
(Test 18)	Burning-point	350–650° F.
(Test 19)	Fixed carbon	2–10 per cent
(Test 21)	Solubility in carbon disulfide	98–100 per cent.
	Non-mineral matter insoluble	0– ½ per cent
	Mineral matter	0– ½ per cent
(Test 22)	Carbenes	0– 1 per cent
(Test 23)	Solubility in 88° petroleum naphtha	80– 99 per cent
(Test 28)	Sulfur	Tr.– 5 per cent
(Test 30)	Oxygen	0– 3 per cent
(Test 33)	Solid paraffins	0– 15 per cent
(Test 34*a*)	Saturated hydrocarbons	30– 90 per cent
(Test 34*b*)	Sulfonation residue	90–100 per cent
(Test 37*e*)	Saponifiable constituents	Tr.– 5 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Table XXXIV includes a few tests on typical residual oils exclusive of cut-back products.

Pressure tars, also known as cracking-still tars have a smaller percentage of hydrogen than the original charging stock, or fraction thereof. Dehydrogenation is one of the steps by which such products are formed. The various steps include dehydrogenation, polymerization, condensation, intermolecular rearrangement, and various combinations of these reactions. Polymerization and condensation, accompanied by dehydrogenation, account for the formation of asphalt in the cracking process. It is of interest to note that asphalt is formed in the cracking process from fractions of non-asphaltic crude oils (e. g., Pennsylvanian), or semi-asphaltic crudes (e. g., Mid-continental) which are more paraffinic than asphaltic, and from which asphalts are not otherwise produced in commercial quantities. The formation of asphalts from paraffin-base oils (i. e., non-asphaltic) is a most striking example of the synthesis of asphalts from paraffin hydrocarbons.[80]

Table XXXV shows the characteristics of residual oils obtained from cracking various fuel oils and topped crudes made by a so-called flashing operation of the cracking process, in which the product is withdrawn from the cracking still and transferred to a vaporizing chamber or zone of reduced pressure. This reduction in pressure causes the vaporization of controlled amounts of the product. The residue withdrawn from the vaporizing chamber is termed

TABLE XXXIV

Characteristics of Typical Residual Oils

Test No.	Test	From Non-Asphaltic Petroleum		From Semi-asphaltic Petroleum								From Asphaltic Petroleum						
		U. S. Appalachian	U. S. Lima-Indiana	U. S. Mid-Continental						U. S. Illinois		Gulf Coast (Texas)	California		Mexican	Trinidad	Venezuela	Colombia
	Physical Characteristics:																	
3	Appearance surface aged 7 days...	D	D	SD	SD	B	SD	B	B	B	B	B	B	B	B	B	B	B
7	Specific gravity at 77° F.....	0.900	0.925	0.910	0.935	0.950	0.960	0 995	0.985	0 925	0.950	0.968	0.965	0.978	0 989	1 000	1.027	0.985
	Mechanical Tests:																	
8a	Specific Engler-viscosity at 140° F.	12.2	15.8	13.0	17.6	21.0	26 5	32 6	28.3	14.2	19.5	35.1	15.2	26.7	38.3	28 0	45.3	40.8
9	Consistency at 77° F.....	0.0	0.0	0 0	0.0	0.0	0.0	0.0	0 0	0.0	0.0	0.0	0.0	1.1	4.2	0.0	0 0	3.5
	Consistency at 32° F.....	1.2	2 9	1.3	3 0	3.4	4.7	5 2	4.5	0.0	3.4	6.1	0.0	6.5	25.2	5 4	20.8	26.2
	Thermal Tests:																	
15a	Fusing-point (K. and S. method) deg. F.....	37	43	25	40	45	50	58	65	35	45	80	30	65	85	55	90	88
15b	Fusing-point (R. and B. method) deg. F.....	45	54 5	35	52	58	60	69	82	43.5	55	94	38.5	76	99	64	102	104
16	Volatile, 500° F., 5 hrs., per cent..	7.8	5.1	4.5	3 0	1.36	2.8	12.25	3.9	7 3	1.4	2.3	14.4	7.3	5.2	4 1	2.2	0.5
17a	Flash-point, deg. F.....	425	462	515	520	516	492	415	502	415	516	463	310	488	465	480	482	550
19	Fixed carbon, per cent.....	2.3	4.2	2.6	4.45	3.65	3 7	4.8	4.35	2.9	3.65	5.7	4.53	6.8	10.5	9 8	9 5	8.2
	Solubility Tests:																	
21	Soluble in carbon disulfide, per cent.	100 0	99.0	99.9	99.9	99.9	99 9	99.8	99.8	99.9	99.9	99.8	99.8	99.65	99.7	99.75	99.8	99.9
	Non-mineral matter insoluble, per cent.....	0.0	0 1	0.1	0.1	0.1	0.1	0.2	0 2	0.1	0.1	0 2	0.2	0.30	0 25	0.20	0.2	0.1
	Mineral matter, per cent.....	0 0	0.0	0.0	0.0	0.0	0 0	0 0	0.0	0.0	0.0	0.0	0 0	0.05	0 05	0.05	0.0	0.0
23	Soluble in 88° petroleum naphtha, per cent.....	98.6	97.1	98 8	98.2	97.9	96.9	90 8	91.7	94.3	97.9	93.2	90.7	84 8	80 6	85 3	82.6	91.7
	Chemical Tests:																	
28	Sulfur, per cent.....	0.0	0.8	....	....	...	0 6	....	....	....	....	2.1	1 5	1.8	4.2	2.9	3.2	2.8
33	Solid paraffins, per cent.....	10.9	7.2	0 3	0.3	0.78	0.9	0 7	1 1	1.6	0.78	0.2	0.2	0.1	0 05	0.0	0.0	0.1
34a	Saturated hydrocarbons, per cent..	85.0	76.2	52.3	70.0	....	53 0	63 2	....	64.6	....	38.0	30.5	....	35.8	36.4	32.5	37.8
34b	Sulfonation residue, per cent.....	98.2	.....	96.0	....	97.5	95.7	....	...	98 0	...	98.5	96.6	....	98 0	95.8	....	....

D = Dull; SD = Slightly dull; B = Bright.

"flashed residue." The yield of pressure tar based on the weight of charging stock depends upon the character of the oil treated. The particular examples given range in yield from 10 to 20 per cent.

TABLE XXXV

ASPHALTS PRODUCED FROM FLASHED RESIDUES

Property	Kentucky*	Midcontinent and West Texas		Midcontinent Cushing§	Midcontinent ‖
		Coating Asphalt†	Asphalt Cement‡		
Specific gravity at 25° C. (77° F.)	1 075	1.065	1.099	1.0867	1.1195
Flash, ° C. (° F.)	231 (448)	.....	.....	171 (340)	177 (350)
Loss at 163° C. (325° F.), per cent	0.58	Trace	1.7	0.765	1.15
Penetration of residue at 25° C. (77° F.), mm	48	4.0	.....	54	49
Softening-point, ° C. (° F.)	53 (127)	126 (259)	55 (131)	54 (129)	51 (123)
Float-test at 50° C. (122° F.) sec	680	.....	829	1210	693
Solubility in CCl_4, per cent	93.8	96.9	.....	91.2	92.7
Solubility in CS_2	99.1	98.6	97.9	98.7	99.8
Paraffin scale	5.3	.....	2.63	3 6	2.17
Fixed carbon	17.2	19.0	20.0	17.8	20.2
Penetration at 25° C. (77° F.), mm	100	5.5	100	97	103
Ductility at 25° C. (77° F.), cm	>122	0.3	125	78	>122
Ductility at 16° C. (60° F.), cm	>122	.....	63	.....	>122

* 100–120 mm. penetration for macadamized roads.
† Coating asphalt, air-blown for 3 hours at 254° C. (490° F.).
‡ Asphalt cement for use in asphalt macadam pavements.
§ 85–100 mm. penetration for macadamized roads.
‖ 100–120 mm. penetration used in building asphalt concrete roads or macadam roads.

Table XXXVI gives a comparison of the properties of the asphalt derived from a cracked Cushing topped crude, by steam-distillation and air-blowing, respectively.

TABLE XXXVI

PROPERTIES OF ASPHALT MADE FROM CUSHING TOPPED CRUDE BY STEAM DISTILLATION AND BY AIR-BLOWING

Property	Residue from Steam Distillation	Air-blown at 232° C. (450° F.)
Flashed residuum, per cent	71.5	70.2
Softening point, ° C. (° F.)	51 (124)	63 (146)
Penetration at 25° C. (77° F.), mm	105	93
Ductility at 25° C. (77° F.), cm	40	11.3
Solubility in CS_2, per cent	100	100
Solubility in CCl_4, per cent	99.3	99.5

Table XXXVII similarly compares the properties of asphalt derived from the flashed residuum from a cracked Pennsylvania kerosene distillate by steam-distillation and air-blowing, respectively.

TABLE XXXVII

PROPERTIES OF ASPHALT MADE FROM PENNSYLVANIA CRUDE BY AIR-BLOWING AND STEAM-DISTILLATION

Property	Cracked Residuum	
	Air-blown	Steam-distilled
Per cent asphalt recovery	5.26	5.09
Specific gravity at 25° C. (77° F.)	1.099	1.116
Penetration at 25° C. (77° F.), mm.	103	105
Softening point (ring-and-ball), ° C. (° F.)	51 (123)	44 (112)
Float test, sec.	381	297
Ductility at 25° C. (77° F.), cm.	125	125
Volatility at 163° C. (325° F.), per cent.	0.67	0.7
Penetration of residue at 25° C. (77° F.), mm.	60	70
Flash point (Cleveland open-cup), ° C. (° F.)	202 (395)	188 (370)
Fire point (Cleveland open-cup), ° C. (° F.)	218 (425)	207 (405)
Solubility in CS_2, per cent.	99.59	99.86
Solubility in CCl_4, per cent.	97.71	98.95
Water	None	None

Such asphalts may be produced to meet specifications for roofing or shingle saturants, coating asphalts for composition roofings, floorings, blowing stock, emulsions, etc. For a given stock, the pressure tar obtained from a cracking coil operated at 250 lb. per square inch will be less asphaltic than a tar obtained from the same unit operated at 750 to 1000 lb. per square inch. In no case is a present-day cracking unit operated under conditions where appreciable quantities of free carbon is formed.

The outstanding features of high temperature-pressure conversion residues are as follows:

(1) Up to a certain point, the higher the temperature and pressure, or the longer the time element, the more asphaltic will be the residue obtained.

(2) Cracking-coal residues of a given fusing-point have a higher specific gravity, ductility and susceptibility to temperature

changes, likewise lower viscosity than equivalent residues produced by direct distillation processes.

(3) Cracking-coil asphalts are less soluble in petroleum naphthas than straight-distilled residues produced from the same crude.

Straight-reduced asphalts from cracking processes resemble coal-tar pitch in certain physical properties, since they have extremely low viscosity when melted and produce cut-backs and primers that are if anything superior to coal-tar products.[31] Incidentally, pressure tars are completely miscible with coal tars and coke-oven tars.[32]

The following figures [33] show the characteristics of residual oils derived from various sources, as well as sundry mixtures:

TABLE XXXVIII

Nature of Oil	Grade of Oil	Specific Gravity at 25° C.	Engler Specific Viscosity at 60° C.	Solubility in Carbon Disulfide, per cent	Insoluble in 86° Baumé Paraffin Naphtha, per cent	Flash-point, Open Cup, deg. Cent.	Loss at 163° C., 5 hours, per cent	Solid Residue of 100 Penetration, per cent	Ductility of Solid Residue at 25° C., cm.
Straight-run residual oil from U. S. non-asphaltic petroleum (paraffin base)...	Light	0.933	13.7	99.9	1.4	190	0.7	56	11
	Medium	0.939	21 4	99 9	2.9	210	0.7	66	13
	Heavy	0 939	31.5	99 6	1.4	238	0.45	68	22
Straight-run residual oil from Arkansas semi-asphaltic petroleum blended with pressure-tar derived from ditto.....	Light	0 980	13 8	99.6	11.9	130	7.9	66	10
	Medium	1 002	23.9	99.6	14.0	125	9.7	67	4
	Heavy	1.013	30.1	98.8	16.4	134	4.5	69	5
Pressure-tar derived from U. S. non-asphaltic and semi-asphaltic petroleums.....	Light	1.030	13.6	99.9	11.8	170	2.7	61	100+
	Medium	1.045	18 9	99 9	12.4	185	2 0	68	100+
	Heavy	1.056	32.3	99 9	14.2	195	1.6	72	100+
Pressure-tar derived from U. S. non-asphaltic and semi-asphaltic petroleum	Light	0.938	10.3	99.6	5.1	165	4.1	65	2
	Medium	0.944	16.1	99.6	6.3	180	1.9	74	2
Mixture of straight-run residual oils derived from Mexican asphaltic petroleum and U. S. non-asphaltic petroleum	Light	0.947	13.2	99 9	5.7	150	5.2	57	21
	Medium	0.959	23.8	99.6	10.5	126	6.1	64	33
Straight-run residual oil from Mexican petroleum.....	Medium	0.963	23.5	99.9	15.6	105	5.6	63	100+
Cut-back residual oil from Mexican petroleum.....	Light	0.961	15.6	99.8	14.9	98	15.2	59	87

BLOWN PETROLEUM ASPHALTS

These are manufactured from residual oils derived from asphaltic, semi-asphaltic or non-asphaltic petroleums, by blowing with air at elevated temperatures.

It has been recognized for many years that petroleum products become changed in their physical properties by treating with oxidizing agents or air. One of the first to report this was Abraham Gesner in 1865,[34] who remarked that:

"Organic substances are oxidized by the atmosphere, and its action promoted by a high temperature. Hot air has therefore been forced through hydrocarbon oil during the process of purification, and in some instances with advantage."

In 1876 W. P. Jenney patented the process of treating sludge oil obtained in refining petroleum with sulfuric acid, with a current of air at a temperature of 250° C.[35] He observed that a resinous substance was produced by the absorption of atmospheric oxygen by the oil.

De Smedt patented a process for oxidizing coal tar with potassium permanganate or permanganic acid at a temperature of 300° F.,[36] also the method of oxidizing petroleum residues in a similar manner,[37] or by distilling petroleum in the presence of an oxidizing agent.[38]

Next Schall [39] discovered that paraffin, vaseline and heavy mineral oils oxidize rapidly and almost completely by introducing a well-divided stream of oxygen at 140–150° C., especially in the presence of insoluble manganese compounds. Dorsett oxidized coal tar by heating with manganese dioxide and ammonium chloride.[40] Baillard oxidized a mixture of petroleum and oleic acid by means of air at 160° C.[41] Busse blew air through a heated mixture of asphalt and vegetable oils in the presence of nitric acid, sulfur, or sulfur dichloride.[42] Salathé blew heated air through melted native asphalt, either alone or in the presence of manganese dioxide or litharge.[43] Schreiber proposed blowing melted asphalt, coal-tar pitch, fatty-acid pitch, wool-fat pitch, etc., with air in the presence of manganese dioxide, with or without the addition of sulfuric acid, and finally adding formaldehyde.[44] Incidentally, it is of interest to note that vaseline, liquid or solid paraffin wax may be converted into

fatty acids by oxidation with potassium permanganate in acid or alkaline solution, or with a mixture of potassium permanganate and sodium hypochlorite, or a mixture of manganese dioxide with hydrochloric or nitric acid.[45]

The first to manufacture blown asphalt on a commercial scale was F. X. Byerley, who obtained a patent in 1894[46] for drawing air through petroleum residues (and especially those derived from Lima, Ohio, crude oil), at temperatures between 400 and 600° F. In this way he obtained "pitches" of variable properties depending upon the temperature and the duration of the blowing process. For the softer grades (fusing under 200° F.), the Lima residuum was blown three days at 400° F., during which 2 per cent of distillate was produced. For the harder grades (fusing at about 400° F.) the residuum was blown four to five days at 500° F., during which between 5 and 6 per cent of distillate was recovered. The product was claimed to be resistant to changes in atmospheric temperature, and to differ from the corresponding steam-distilled asphalt by being readily soluble in petroleum benzine or naphtha. Byerley marketed the product under the name of "byerlite." Air under suction was passed through a 6000 gal. still of the oil, at the rate of 450 cu. ft. per minute. Ohio petroleum residue of 21 to 27° Baumé, also Texas Gulf-coast residue of 12 to 15 Baumé were first used for this purpose.

J. W. Hayward[47] and also G. F. and G. C. K. Culmer[48] obtained patents for a similar process, according to which a mixture of petroleum residue and refined Trinidad asphalt or gilsonite is heated to 193° C. and blown for forty hours at the rate of 15 to 30 cu. ft. air per minute, per ton asphalt. After a time the external source of heat was removed, since it was found that the temperature of the residue increased spontaneously, due to the chemical changes induced by the action of the air. It was also found that the oxidation progressed very rapidly at first, and then more slowly, as it approached the end of the process.

According to present practice, the residual oil is blown at 525 to 575° F. at the rate of 30 to 50 cu. ft. air per minute, per ton of asphalt, for a period of five to twelve hours. It is found unnecessary to use any of the catalyzers referred to in the earlier patents. The air may either be blown through the still under pressure, or

else sucked through the still by subjecting the contents of the still to a partial vacuum (up to 20 in. mercury). There are proponents of both systems. The loss depends largely upon the amount of volatile matter contained in the residual oil, and varies from practically 0 up to 10 per cent of the weight of the charge, depending of course upon the extent the product is blown. Water and carbon dioxide are also given off. It is contended that the vacuum process removes the oily and greasy matters from the residual oil and forms a brighter and cleaner looking blown asphalt. The more asphaltic the crude from which the residual oil was derived, the better will be the quality of the blown product, and the shorter the duration of the blowing process. Residual oils derived from semi-asphaltic and non-asphaltic petroleums must be blown longer to obtain a product of the same fusing-point, and the blown asphalt is apt to appear quite oily and greasy. Non-asphaltic petroleums will yield a good grade of blown asphalt if first subjected to a high temperature and pressure, which serve to convert saturated hydrocarbons into polymerized unsaturated compounds.[49] Blown asphalts may likewise be produced from naphthenic residual oils or naphthenic derivatives i.e., non-asphaltic and non-paraffinic in character) obtained by extracting petroleum with SO_3, aniline, phenol, etc.[50]

The best blown asphalts are produced from the following petroleums, viz.: U. S. Gulf-coast field (Texas); Mexican heavy viscous crudes of 8 to 15° Baumé obtained from the Panuco and Topila regions (20 to 30 miles southwest of Tampico), also from the Ebano region (40 miles west of Tampico); Venezuela crudes from the Lagunillas, La Roza and Mene Grande fields; Colombian crudes from the Barranca-Bermeja region; likewise from Trinidad crudes.

Mid-continental (U. S.) semi-asphaltic petroleums form residual oils which when blown at 450 to 475° F. for nine to ten hours produce blown asphalts having a fusing-point of 350 to 400° F. (R. and B. method) and without the formation of "free carbon." If a small proportion of gilsonite is added to the mixture, the time of blowing will be increased, but the product will be improved materially in the following respects: it will be harder, have a higher gloss, greater cohesiveness, greater ductility, lower susceptibility to temperature changes, and improved weather-re-

sistance. The use of grahamite does not give as satisfactory results, since the product will be lacking in ductility.

California residual oils produced from asphaltic petroleums when blown at 425 to 450° F. for twelve to eighteen hours will produce blown asphalts having a fusing-point of 175 to 190° F. (R. and B. method). If the temperature exceeds 530° F. free carbon will be formed. Blown California asphalts have a good gloss and are considerably harder than Mid-continental, Gulf-coast (Texas), Mexican, Venezuelan, Colombian and Trinidad products. They are also more susceptible to temperature changes.

Texas residual oils derived from asphaltic petroleum produce excellent blown asphalts that are glossy, resistant to temperature changes, free from oily or greasy constituents, highly ductile, and are excellent weather-resistants.

Mexican, Venezuelan, Colombian and Trinidad asphaltic petroleums produce residual oils which behave similarly on blowing. Such residual oils having a fusing-point of 90 to 100° F. (R. and B. method) when blown at 450 to 500° F., for six and one half to ten hours, at the rate of 40 cu. ft. air per minute, per ton, will produce blown asphalts having a fusing-point of 220 to 235° F. (R. and B. method) and a penetration at 77° F. of 15 to 17. The resultant asphalts have a good gloss, are free from oily constituents, moderately ductile and fairly resistant to temperature changes.

A method has been patented for blowing a mixture of topped Mexican residuum (8 to 10° Bé.) with topped Mid-continental residuum (12 to 14° Bé.) derived from Illinois semi-asphaltic petroleum, at 450 to 500° F., which is claimed to result in a product of greater ductility than when Mid-continental residuum is used alone.[51]

Various catalyzers and oxidizing agents have been proposed for augmenting the air-blowing process, including: manganese dioxide;[52] manganese dioxide and nitric acid;[53] finely powdered limestone;[54] caustic soda or sodium carbonate;[55] bentonite or finely powdered coke;[56] sulfur;[57] sulfuric acid with or without the addition of metallic persulfates or perborates;[58] boric acid, phosphorus acid or arsenious acid;[59] potassium chlorate;[60] chlorides or sulfates of zinc, iron, copper or antimony;[61] basic acetylacetonate of manganese, cerium, nickel, cobalt or zinc;[62] copper, iron, manganese or cobalt soaps;[63] as well as the various agents referred to previously. It

has been proposed that any excess catalyzer be removed after the blowing operation by boiling the product with dilute hydrochloric acid and washing with hot water.[64]

The use of various gases has been suggested for blowing through the melted asphalt, including: air alone; air under superatmospheric pressure;[65] air followed by a mixture of air and steam in varying proportions under atmospheric or reduced pressure;[66] carbon dioxide with or without air;[67] ozone;[68] a mixture of air or oxygen with NO_2 or SO_2;[69] carbon monoxide activated by a catalyst such as nickel or palladium, or by first heating the asphalt with cerium oxide or tin oxide and blowing as aforesaid;[70] air containing up to 11½ per cent chlorine;[71] chlorine followed by carbon dioxide;[72] bromine or boron fluoride; air and halogens in the presence of sulfur;[73] etc.

It has been shown that the use of air containing up to 11½ per cent of chlorine for blowing asphalts maintained at 475° F. speeds up the process, and that chlorine is both absorbed and combined chemically with the asphalt. The fusing-point is increased more rapidly, and the ductility and penetration are decreased more rapidly than when air alone is used. Thus, with a residual oil having a fusing-point of 86° F. (R. and B.) and a penetration of 253 at 77° F. (50 grams in 5 seconds), the following relative blowing times were required:

	To increase the Fusing-point to 200° F. (R. & B. method)	To decrease the Penetration (50 g./5 sec./ 77° F.) to 20	To decrease the Ductility at 77° F. to 10 cm.
Air alone	22 hrs.	18 hrs.	19 hrs.
Air with 1.3 per cent chlorine	18 hrs.	15 hrs.	14 hrs.
Air with 6.0 per cent chlorine	8 hrs.	5 hrs.	5½ hrs.
Air with 11.2 per cent chlorine	5 hrs.	3 hrs.	3½ hrs.

The blowing operation may either be intermittent or continuous.[74] In the intermittent or batch process the apparatus consists essentially of an open, or preferably closed cylindrical still in which the air or other gas is introduced through a series of perforated pipes at the bottom. Various means have been suggested to subdivide the gas into fine streams or jets,[75] or to accomplish a more

intimate mixing of the elements by rapidly agitating the asphalt in a closed vessel through which a current of air is passed.[76] Continuous processes involve the following expedients: circulating the asphalt through a horizontal still divided into compartments into which air is blown individually;[77] circulating the asphalt through a series of stills into each of which air is blown;[78] passing the asphalt in conjunction with a stream of air through a tube-heater;[79] flowing or spraying the melted asphalt through a chamber in which a counter-current of air is passed.[80]

Various mechanical expedients have been suggested to facilitate the blowing process, including the following. To conserve fuel, the freshly distilled residual oil, before it is permitted to cool, is blown in a heat-insulated still without the application of external heat.[81] Constant temperature is maintained during the blowing operation by a thermostatically controlled cooling device.[82] To prevent further oxidation of the blown product while still at a high temperature, it is maintained in contact with an inert gas.[83] Devices for condensing and returning to the still any vapors that are evolved during the blowing process.[84] Recirculating the air as well as any evolved vapors through the charge in a closed system.[85] The blown product may be "stabilized" by maintaining at a high temperature for 24 hours.[86]

The use of different mixtures has been suggested for the purpose of improving the characteristics of the finished product, in the way of imparting rubber-like properties, greater toughness, increasing its resistance to temperature changes, augmenting its weather-resistance, etc., including the following: a mixture of semi-asphaltic residual oil and fatty-acid pitch, with or without gilsonite;[87] a mixture of residual oil, pine tar and rubber;[88] a mixture of residual oil or residual asphalt with vegetable drying oil;[89] a mixture of residual asphalt with cylinder oil stock;[90] a mixture of gilsonite with cylinder oil stock;[91] asphalt mixed with the high-boiling point, SO_3 insoluble residue derived from the Edeleanu extraction process;[92] a mixture of asphalt with pressure tar (cracking-still residue);[93] a mixture of sludge asphalt neutralized with magnesium oxide;[94] blowing an aqueous-clay dispersion of residual oil with metallic driers at a comparatively low temperature.[95]

Blown asphalts vary in consistency from semi-liquids to moder-

ately hard solids at room temperature. They are marketed under various proprietary names such as Byerlite, Sarco, Hydrolene, Texaco, Parolite, Korite, Stanolind, Obispo, Ebano, etc.

The advantages of "blowing" over the steam-distillation process are as follows:

(1) The yield of asphaltic residue from the blowing process is much greater than when steam distilled.

(2) Blown petroleum asphalts are less susceptible to temperature changes than steam distilled products, and in addition acquire a certain amount of elasticity and resilience, usually termed "rubber-like" properties. Upon comparing a residual asphalt with a blown product of the same fusing-point, it will be found that the latter is considerably softer, as evidenced by its consistency or penetration. Conversely, upon comparing a residual asphalt with a blown product of the same hardness or penetration, the fusing-point of the latter will be found to be considerably higher.

(3) In many cases it is possible by blowing to obtain a residue of better quality than if the steam-distillation process were used, and in fact the blowing process renders many crude petroleums available which could not otherwise be used for preparing *high* fusing-point asphalts. Non-asphaltic petroleum will produce fairly good asphalts when blown, whereas the same crude will produce worthless residual asphalts by the steam distillation process.

(4) It is easier to control the "grade" of asphalt by blowing. As previously noted, the progress of blowing is more rapid at the start of the process than towards its conclusion. In other words, the blown asphalt is said to "come to grade" very slowly. With steam distillation, the alteration is much more marked at the end of the distillation process, so that the steam-distilled asphalts "come to grade" very rapidly. In attempting, therefore, to produce a residual asphalt of a definite fusing-point or hardness, the blowing process is preferable, since it may be controlled to better advantage.

Figure 129 shows the effect of prolonging the blowing process (at 425° F.) on the fusing-point (Ring and Ball method, Test 15*b*), and penetration (needle penetrometer, test 9*b*) of a topped mixed-base petroleum.[96] The ductility-fusing-point curve is almost a straight line for residual asphalts derived from the same crude oil, whereas in the case of blown asphalts a decided break or change in direction is found to occur.[97]

Figure 130 illustrates the consistency, tensile strength (multiplied by 10) and ductility curves of a typical blown petroleum as-

phalt produced from mid-continental petroleum, having a fusing-point of 127° F. (K. and S. method).

The care with which blown asphalts are prepared largely influences their physical characteristics. When made from improper

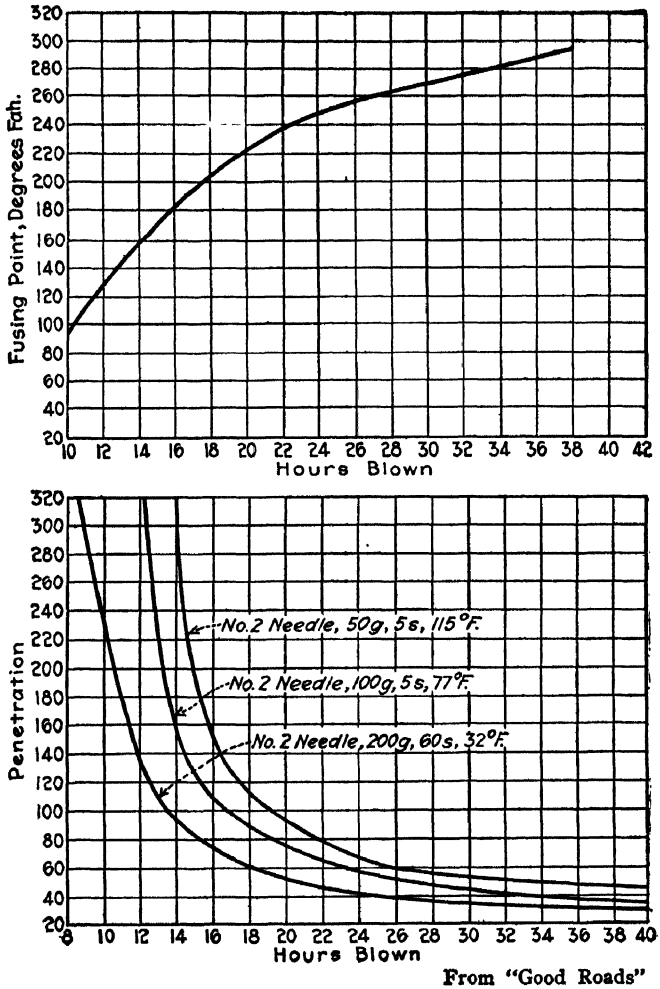


FIG. 129.—Effect of Blowing on the Fusing-point and Hardness of Petroleum Asphalt.

crudes or by careless treatment, blown asphalts are apt to have certain defects, viz.:

(1) When made from non-asphaltic or semi-asphaltic petroleums, they are likely to present a "greasy" surface, and especially on standing a few days, due to the partial separation of vaseline- or paraffin-like bodies. Blown asphalts made from *asphaltic* crudes

do not behave in this manner. The oily constituents and wax may be separated by first treating the crude petroleum with a selective solvent, as for example propane,[98] or liquid SO_3, aniline, phenol, etc.,[99] distilling off the solvent and blowing the residue in the usual manner. It is also claimed that these objectionable constituents may be eliminated if the asphalt is blown with air containing ammonia gas.[100]

(2) Blown asphalts, and especially those of high fusing-point, have the disadvantage of being "short," or in other words, they lack ductility. By carefully regulating the process, this defect may be minimized, particularly if the asphalt is subjected to a *moderate* amount of blowing. It is a fact, however, that the longer the blow-

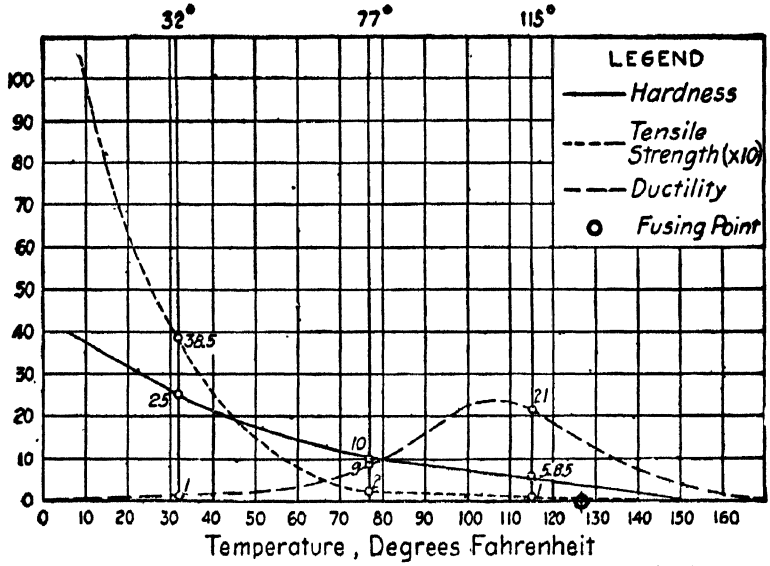


FIG. 130.—Physical Characteristics of Blown Petroleum Asphalt.

ing is continued, the less ductile will be the asphalt. The ductility of the product may be decidedly improved by adding to the base material a proportion of the product derived from the Edeleanu extraction process (i. e., hydrocarbons soluble in liquid SO_2) and blowing the mixture.[101]

(3) Asphalts when over-blown, or blown at too high a temperature, show a separation of non-mineral matter insoluble in carbon disulfide, and a large percentage of carbenes. The former may readily be detected under the microscope (see Test 2*b*), also in certain aggravated cases by the eye, by presenting a dull surface upon being disturbed (see Test 2*a*).

When the blown asphalts first appeared on the market, they unfortunately did not enjoy a good repute, but their quality has

improved to such an extent, that blown asphalts may now be procured of almost any fusing-point up to 400° F., which are not only more resistant to temperature changes, but are at the same time as ductile as any unblown product of the same "grade" (i. e., fusing-point or hardness at 77° F.).

In general, blown asphalts comply with the following characteristics:

(Test 1)	Color in mass	Black
(Test 2*a*)	Homogeneity to the eye at room temperature	Uniform to gritty
(Test 2*b*)	Homogeneity under the microscope	Uniform to lumpy
(Test 3)	Appearance surface aged indoors one week	Bright to dull and greasy
(Test 4)	Fracture	Soft grades do not show a fracture, hard grades present a conchoidal fracture
(Test 5)	Lustre	Bright to dull
(Test 6)	Streak on porcelain	Brownish black to black
(Test 7)	Specific gravity at 77° F.	0.90–1.07
(Test 9*b*)	Penetration at 77° F.	25–200
(Test 9*c*)	Consistency at 77° F.	2–30
(Test 9*d*)	Susceptibility index	8–40
(Test 10)	Ductility	Variable
(Test 11)	Tensile strength	Variable
(Test 15*a*)	Fusing-point (K. and S. method)	80–400° F.
(Test 15*b*)	Fusing-point (R. and B. method)	100–425° F.
(Test 16)	Volatile matter, 500° F. in 5 hours	1–12 per cent
(Test 17*a*)	Flash-point	350–550° F.
(Test 18)	Burning-point	400–650° F.
(Test 19)	Fixed carbon	5– 20 per cent
(Test 21)	Solubility in carbon disulfide	95–100 per cent
	Non-mineral matter insoluble	0–5 per cent
	Mineral matter	0–½ per cent
(Test 22)	Carbenes	0–10 per cent
(Test 23)	Solubility in 88° petroleum naphtha	50–90 per cent
(Test 25)	Water	Absent
(Test 28)	Sulfur	Tr.– 7.5 per cent
(Test 30)	Oxygen	2– 5 per cent
(Test 33)	Solid paraffins	0– 10 per cent
(Test 34*a*)	Saturated hydrocarbons	30– 75 per cent
(Test 34*b*)	Sulfonation residue	90–100 per cent
(Test 37*e*)	Saponifiable constituents	Tr.– 2 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Figure 131, plotted by the author from hundreds of determinations, shows the relation between the specific gravity and fusing-point of residual oils, blown asphalts and residual asphalts.

Residual oils range in specific gravity from 0.85 to 1.07 at 77° F., and in fusing-point (K. and S. method) from 20 to 100° F.;

blown asphalts range in specific gravity from 0.90 to 1.07, and in fusing-point from 80 to 400° F.; residual asphalts range in specific gravity from 1.00 to 1.17 and in fusing-point from 80 to 225° F. The chart shows that residual oils verge into the residual and blown asphalts respectively, and it also graphically illustrates the difference between the residual and blown asphalts. It will be observed that the effect of blowing is to increase the fusing-point and decrease the specific gravity of the product.

Another method for distinguishing between blown and residual asphalts, devised by the author, consists in finding their hardness by means of the consistometer (Test 9*c*) at a temperature *exactly*

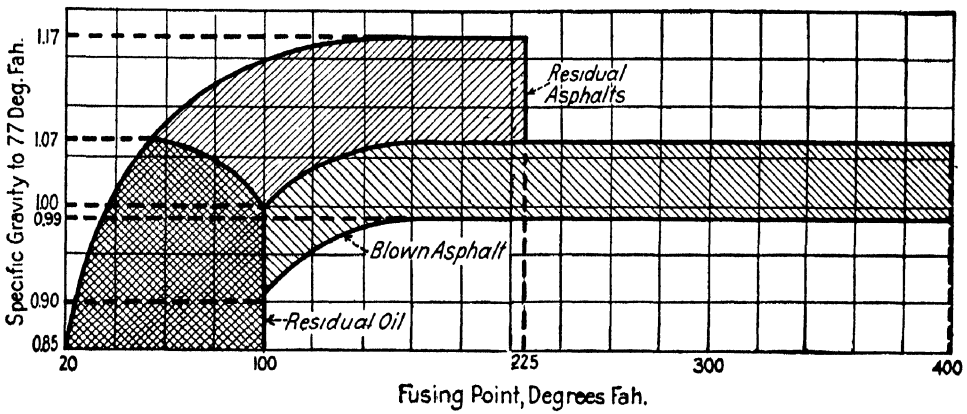


FIG. 131.—Relation between the Specific Gravity and Fusing-point of Residual Oils, Blown Petroleum Asphalts and Residual Asphalts.

50° F. *lower* than their fusing-point by the K. and S. method. Blown asphalts show a hardness of less than 15, whereas residual asphalts show a hardness greater than 15 under these conditions. This is illustrated by the examples given in Table XXXIX.

It is of interest to note in connection with the foregoing figures, that for any particular crude, the hardness at the "fusing-point less 50° F." decreases with the extent of the blowing, but remains practically constant in the case of the residual asphalts, regardless of the extent to which the distillation may have been carried.

Blown asphalts, due to their greater softness, and correspondingly large proportion of "life-giving" constituents, are better weather-resistants than residual asphalts derived from the same crude, having the same fusing-point and showing the same propor-

TABLE XXXIX

	Fusing-point, (Test 15*a*)	Hardness (Test 9*c*) at 50° F. below the Fusing-point
Blown Petroleum asphalts:		
Lima, grade 120	91.5° F.	Hd. at 41.5° F. = 11.2
Lima, grade 150	155	Hd. at 105.0° F. = 10.1
Lima, grade 185	214	Hd. at 164.0° F. = 7.8
Lima, grade 285	271.5	Hd. at 221.5° F. = 6.7
Mid-continental, grade 115	100	Hd. at 50.0° F. = 11 9
Mid-continental, grade 180	164	Hd. at 114.0° F. = 10.3
Mid-continental, grade 215	215	Hd. at 165.0° F. = 8.4
Residual asphalts:		
California, grade *E*	90	Hd. at 40.0° F. = 21.5
California, grade *DE*	108	Hd. at 58.0° F. = 22.2
California, grade *D*	124	Hd. at 74.0° F. = 22.2
California, grade *C*	146	Hd. at 96.0° F. = 22.9
California, grade *CB*	168.5	Hd. at 118.5° F. = 23.5
California, grade *B*	209	Hd. at 159.0° F. = 22.9
Mid-continental, grade 140	119.5	Hd. at 69.5° F. = 21.5
Mid-continental, grade 180	158	Hd. at 108.0° F. = 21.5

tion of volatile matter. They are about equal in weather-resistance to residual asphalts of the same hardness prepared from the same crude, and far superior to sludge asphalts regardless of their hardness or fusing-point. It is a mooted question whether the native asphalts or the blown asphalts excel in weather-resisting properties, and to which in the author's opinion no categorical answer may be given.

It has been observed that blown asphalts have a higher saponification value than residual asphalts, upon comparing grades of the same fusing-points.[102] Three samples of the same product tested as follows:

	Fusing-point (K. and S.) (Test 15*a*)	Acid Value (Test 37*a*)	Saponification Value (Test 37*d*)
Sample No. 1	70° C.	0.03	12.05
Sample No. 2	100° C.	0.03	38.9
Sample No. 3	150° C.	0.03	42.0

It is concluded that the saponification value of asphalts is increased by blowing, in a manner similar to that observed with

paraffin waxes, etc., and that ester-like bodies and anhydrides are produced during the blowing process. On the other hand Edmund Graefe [103] ascribes the high saponification values (some of which he found to be as high as 60) to the presence of sulfuric acid in the blown asphalt. He points out that all asphalts normally carry sulfur in combination, and that in some of them, e. g., Mexican asphalts, the sulfur is loosely combined with the associated hydrocarbons, so that during the blowing process this loosely combined sulfur unites with the oxygen of the air introduced into the heated asphalt, forming sulfuric acid. He predicts that if this sulfuric acid remains in the asphalt, it is apt to cause its disintegration upon exposure to the elements. Graefe also reports that the oily distillate condensed during the blowing process will combine with strong alkalies, which when neutralized with mineral acid forms a light colored, stringy resinous mass. On the other hand, it has been claimed that the weather-resistance of blown asphalts may be improved by treating the product with a 10 per cent solution of hydrochloric acid.[104]

Table XL includes the results obtained by the author upon examining representative blown asphalts derived from different crudes and blown to different extents.

SULFURIZED ASPHALTS

The process of treating asphalt with sulfur was first disclosed by A. G. Day [105] and subsequently by J. A. Dubbs [106] who heated Pennsylvania, Lima and Ohio residuums with 20 to 25 per cent of sulfur, at a temperature somewhat below the boiling-point of sulfur, until the evolution of gas ceased. The resulting product is very similar in its physical properties to blown asphalt, being only slightly susceptible to temperature changes, but it is still further lacking in ductility. Thirty years ago, asphalt treated in this manner was exploited under the name "Pittsburgh Flux." This was before blown asphalts appeared on the market, which on account of their smaller cost of production, soon displaced the sulfurized product.

Processes have been described involving the vulcanization with sulfur of the following classes of bituminous products: native asphalts;[107] native asphalt (e. g. Trinidad) mixed with vegetable oils;[108] native asphalt (e. g. Trinidad) fluxed with fatty-acid

TABLE XLI.—CHARACTERISTICS OF TYPICAL RESIDUAL ASPHALTS

No.	Test	From Semi-asphaltic Petroleum				From Asphaltic Petroleum													
		Mid-continental				Mexican			California					Gulf-coast		Trinidad		Venezuela		Japan
	Physical Characteristics:																		
2a	Homogeneity to eye at 77° F.	Homo.	Homo.	Homo.	Non-hom.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.	Non-hom.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.
2b	Homogeneity under microscope	Homo.	Homo.	Homo.	Lumpy	Gritty	Homo.	Gritty	Homo.	Homo.	Homo.	Gritty	Lumpy	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.
3	Appearing surface, aged 7 days	Dull	Dull	Dull	Dull	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright
4	Fracture	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	Conch.	.....	Conch.	Conch.	Conch.	.....
5	Lustre	Bright	Bright	Bright	Sl. dull	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	Bright	.....	Bright	Bright	Bright	.....
6	Streak	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black	Black
7	Specific gravity at 77° F.	1.050	1.078	1.119	1.145	1.015	1.024	1.036	1.065	1.095	1.113	1.127	1.158	1.022	1.033	1.095	1.120	1.082	1.105	1.050
	Mechanical Tests:																			
9b	Penetration at 115° F.	88	45	12.5	6	132	99	84	70	28	11.5	8	7	48	40	51	12	78	46	Soft
	Penetration at 77° F.	37	21	3.5	1	32	24	22	24	15	3	½	0	19	9	20	2	26	17	93
	Penetration at 32° F.	9.5	5	0	0	12	8	7	10	6	0	0	0	14	10	12	0	7	0	16
9c	Consistency at 115° F.	5.1	9.6	27.1	47.9	3.1	4.5	6.5	6.8	13.9	28.8	38.0	45.1	8.2	10.4	8.8	27.2	6.0	9.4	2.8
	Consistency at 77° F.	15.2	23.5	58.5	88.8	17.2	21.5	22.9	21.5	29.0	63.0	89.2	98.0	25.2	37.0	24.0	70.2	20.2	26.6	6.1
	Consistency at 32° F.	66.9	81.4	107.0	>100	60.4	68.0	73.0	64.6	78.3	>100	>100	>100	56.3	65.1	88.7	>100	72.1	>100	51.0
9d	Susceptibility index	51.7	50.5	48.0	> 45	47.3	47.8	46.5	46.6	44.0	> 42	> 40	> 45	32.0	29.2	43.3	> 45	46.5	> 50	41.9
10b	Ductility in cms. at 115° F.	37	64.5	12	0	45	42.5	46.5	57	12.5	8.0	0	0	50	32	70	3.5	42	22	0
	Ductility in cms. at 77° F.	19	10	0	0	8	3.5	3	9	1	0	0	0	28	15	22	0	20	15	100
	Ductility in cms. at 32° F.	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0	12
11	Tensile strength in kg. at 115° F.	0.1	0.35	2.0	5.5	0.4	0.8	0.75	0.6	1.65	3.95	3.4	4.25	3.8	5.5	0.4	4.85	0.8	1.6	.....
	Tensile strength in kg. at 77° F.	2.7	4.2	6.0	4.0	2.8	4.0	5.0	3.5	6.2	5.0	4.0	4.2	5.2	7.0	2.8	5.5	4.1	6.2	.....
	Tensile strength in kg. at 32° F.	15.0	9.5	7.0	5.0	8.5	11.0	10.5	10.0	12.0	8.0	5.2	3.7	12.0	18.0	8.7	10.2	10.0	13.4	.....
	Thermal Tests:																			
15a	Fusing-point, deg. F. (K. and S. method)	119.5	142	167	205	121	133	143	124	146	168.5	209	218	150	187	115	187	142	180	115
15b	Fusing-point, deg. F. (R. and B. method)	131.5	159	184	227	141.5	154	162	142	163	188	220	229.5	167	205	132	210	160	195	133
16	Volatile, 500° F. in 5 hrs., per cent	0.78	0.50	1.00	0.71	0.76	0.50	0.47	3.08	2.44	3.99	3.72	2.12	0.63	0.37	3.08	2.75	1.41	0.75	6.2
17a	Flash-point, deg. F.	525	532	575	595	520	555	547	500	525	535	545	563	530	552	528	561	487	525	420
19	Fixed carbon, per cent	13.8	18.5	23.1	33.3	28.9	30.6	32.3	24.8	30.2	34.0	37.0	39.7	25.7	33.5	29.1	38.4	32.0	36.7	17.3
	Solubility Tests:																			
21	Soluble in carbon disulfide	99.25	99.03	96.45	91.02	97.50	98.22	97.95	98.93	98.67	98.64	98.12	86.20	99.5	98.3	99.35	98.39	98.7	98.5	99.2
	Non-mineral matter insoluble	0.50	0.70	3.12	8.20	2.15	1.33	1.80	1.07	0.90	1.10	1.60	13.48	0.2	0.3	0.48	1.28	0.1	0.2	0.4
	Mineral matter	0.25	0.27	0.43	0.78	0.35	0.45	0.25	0.30	0.43	0.26	0.28	0.32	0.3	0.4	0.17	0.33	0.2	0.3	0.4
22	Carbenes	1.8	3.0	5.0	12.3	0.6	1.82	2.2	1.5	2.5	4.2	5.6	28.2	0.2	0.3	0.3	0.8	0.1	0.2	0.1
23	Soluble in 88° petroleum naphtha	82.5	80.3	72.3	61.0	76.8	68.9	62.0	71.2	64.1	52.3	45.2	35.7	72.2	66.7	78.0	66.6	68.5	65.0	79.0
	Chemical Tests:																			
28	Sulfur	0.95	.....	1.3	0.6	6.4	4.2	5.8	1.2	1.4	.....	0.8	.....	0.75	0.61	2.8	2.2	2.5	2.0	0.6
33	Solid paraffins	1.3	1.25	0.8	0.5	2.6	1.7	1.4	Tr.	Tr.	0.0	0.0	0.0	0.2	0.1	0.0	0.0	0.0	0.0	0.1
34a	Saturated hydrocarbons	.....	60.7	.....	70.1	38.5	42.9	30.3	.....	30.6	.....	35.8	37.0	38.0	42.3	24.0	28.3	27.5	32.1	35.2
34b	Sulfonation residue	.....	95.0	.....	.....	97.5	.....	.....	.....	.....	.....	98.0	.....	.....	.....	.....	96.4	97.0	98.6	.....

To face page 452

pitch;[109] residual asphalts;[110] residual asphalt mixed with vegetable oils;[111] residual asphalt fluxed with fatty-acid pitch;[112] residual asphalt in combination with aluminium oleate or stearate;[113] sludge asphalt;[114] wurtzilite asphalt;[115] gilsonite mixed with vegetable oils;[116] gilsonite fluxed with fatty-acid pitch;[117] asphalts fluxed with resins;[118] mixture of asphalt, rosin, rubber and vegetable oil;[119] boiling an aqueous dispersion of asphalt with sulfur.[120]

In place of sulfur alone, the use of the following agents has been proposed for vulcanizing asphalts and mixtures containing the same: mixture of sulfur and FeS;[121] mixture of sulfur and SO_2 with a catalyst (e. g., a sulfide or oxide of P, As, Sb, Sn, Mo, V, or W);[122] sulfur and lead peroxide;[123] sulfur and calcium oxychloride;[124] sulfur and sulfuric acid;[125] sulfuric acid alone;[126] spent iron oxide (containing sulfur) obtained as a by-product in desulfurizing coal-gas;[127] antimony sulfide, with or without sulfur;[128] sulfur in the presence of chlorine;[129] sulfur dichloride;[130] sulfurylchloride or pyrosulfurylchloride;[131] chlorine in the presence of a "chlorine carrier" (e. g. hexachlorethane);[132] cyanide residues;[133] etc.

RESIDUAL ASPHALTS

As stated previously, these are derived from the steam distillation of semi-asphaltic or asphaltic petroleums.[134] Non-asphaltic petroleums are unsuitable for manufacturing residual asphalts, although processes have been devised based upon their use.[135] The distillation is continued until the residual asphalt reaches the desired "grade." The temperature of the residue in the still is carefully observed, and under no circumstances allowed to exceed 750 to 800° F., otherwise excessive decomposition and cracking of the hydrocarbons will take place, and result in the production of an inferior product.[136] This is especially liable to be the case if a residual asphalt of hard consistency and high fusing-point is to be produced. Mexican petroleums are more susceptible to overheating, and great care must be taken not to allow the residue in the still to exceed a temperature of 575 to 625° F. The distillation must not proceed too rapidly, otherwise a certain amount of cracking will take place, and the asphalt will have too low a flash-point and contain a certain amount of free carbon. In the early days of the

industry, the residual asphalts were carelessly manufactured, without suitable temperature control, and as a result they soon fell into disrepute. At the present time, residual asphalts are being marketed of excellent quality, including products fusing as high as 225° F., with a hardness in the neighborhood of 100 on the consistometer scale (Test 9*c*), but this is due solely to the better methods of control. Other things being equal, it is not as easy to produce a residual asphalt as a blown product of a given high fusing-point.

A process [137] for removing undesirable constituents of a paraffin- or vaseline-like character from residual asphalts (12 to 24° Baumé) derived from a mixed-base petroleum, consists in introducing large quantities of low-pressure steam superheated to 600 to 700° F., through a charge of the asphalt heated in a still to 450 to 700° F., or by distilling the asphalt under vacuum.[138] Any free carbon may be removed from the topped petroleum or pressure tar or residual oil prior to the final distillation, by precipitation,[139] or by centrifuging,[140] or by acid-treatment.[141] The temperature may be suitably controlled during the distillation process by supplying heat through the medium of a highly-heated mass of so-called "heat-carrier" (e. g. asphalt, fused metallic salts, molten lead or tin, etc.).[142]

Carelessly prepared residual asphalts may be detected by:

(1) Lack of homogeneity (Test 2). This may be due either to over-heating or because the distillation has been continued too far.

(2) The surface of the material assuming a "greasy" appearance on aging. This is due to the use of crudes containing too large a proportion of paraffin- or vaseline-hydrocarbons, which have not been removed during the distillation process.

(3) The presence of too large a percentage of volatile matter (Test 16) or too low a flash-point (Test 17), due to the distillation not having been carried far enough, or at a temperature sufficiently high to remove the low boiling-point constituents.

(4) A large percentage of non-mineral matter insoluble in carbon disulfide (Test 21). This is due either to overheating or to the distillation having been carried too far. Carefully prepared residual asphalts should not contain more than 5 per cent.

(5) The presence of carbenes (Test 22), which are produced by overheating. Carefully prepared residual asphalts should not contain more than 2 per cent.

Residual asphalts made from semi-asphaltic petroleum are not as susceptible to overheating as those derived from purely asphaltic petroleum. In both cases the percentage of asphalt recovered in the distillation process is *greater* than that contained in the original petroleum, due to the fact that the heavy lubricating oils are *polymerized or condensed* under the influence of heat into substances resembling asphalt. In a purely asphaltic petroleum, the quantity of residual asphalt formed during the distillation process is proportionately *less* than in the case of mixed-base petroleum, but the residual asphalt in the former case will not show a greasy surface on aging, no matter how carelessly it may have been distilled.

The effect of continued steam-distillation on the fusing-point, specific gravity, specific viscosity, float test, penetration, fixed carbon and percentage insoluble in 88° petroleum naphtha, for Mexican, Californian and Texas residual asphalts has been described by B. A. Anderton.[148]

Residual asphalts in general comply with the following characteristics:

(Test 1)	Color in mass	Black
(Test 2)	Homogeneity	Variable
(Test 3)	Appearance surface aged indoors 1 week	Variable
(Test 4)	Fracture	Conchoidal in the case of hard residual asphalts
(Test 5)	Lustre	Variable
(Test 6)	Streak on porcelain	Black
(Test 7)	Specific gravity at 77° F	1.00–1.17
(Test 9*b*)	Penetration at 77° F	150–0
(Test 9*c*)	Consistency at 77° F	5–100
(Test 9*d*)	Susceptibility index	40–60
(Test 10)	Ductility at 77° F	Variable
(Test 11)	Tensile strength at 77° F	0.5–10.0
(Test 15*a*)	Fusing-point (K. and S. method)	80–225° F.
(Test 15*b*)	Fusing-point (R. and B. method)	100–250° F.
(Test 16)	Volatile matter	Variable
(Test 17)	Flash-point	400–600° F.
(Test 18)	Burning-point	450–700° F.
(Test 19)	Fixed carbon	5– 40 per cent
(Test 21)	Soluble in carbon disulfide	85–100 per cent
	Non-mineral matter insoluble	0– 15 per cent
	Mineral matter	0– 1 per cent
(Test 22)	Carbenes	0– 30 per cent
(Test 23)	Solubility in 88° petroleum naphtha	25– 85 per cent
(Test 28)	Sulfur	Tr.– 10 per cent
(Test 29)	Nitrogen	Tr.– 1.0 per cent
(Test 30)	Oxygen	0– 2½ per cent
(Test 32)	Naphthalene	None
(Test 33)	Solid paraffins	0– 10 per cent

(Test 34*a*)	Saturated hydrocarbons	25– 75 per cent
(Test 34*b*)	Sulfonation residue	90–100 per cent
(Test 37*e*)	Saponifiable constituents	0– 2 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Residual asphalts are characterized by the following features:

(1) Their comparatively high specific gravity, serving to distinguish them from blown asphalts.

(2) Their greater hardness or consistency at 77° F. for a given fusing-point, which also distinguishes them from blown asphalts.

(3) The greater tensile strength at 77° F. for a given fusing-point, which similarly distinguishes them from blown asphalts.

(4) Their lower fusing-point serves to distinguish them from the asphaltites.

(5) The susceptibility index is very much higher than blown asphalts. In the case of residual asphalts the susceptibility index is greater than 40, whereas with blown asphalts it is always less than 40.

(6) The volatile matter, which for a given fusing-point is lower than that contained in the crude native asphalts.

(7) The flash-point, which for a given fusing-point is higher than that of the crude native asphalts.

(8) The fixed carbon, which for a given fusing-point is greater than that of blown asphalts.

(9) The mineral matter, which runs well within 1 per cent and serves to distinguish residual asphalts from most of the native asphalts.

(10) Carbenes when present in percentages in excess of 5, serve to distinguish them from native asphalts.

(11) Solid paraffins when present serve to distinguish residual asphalts from native asphalts, although this test is not infallible, for as pointed out previously, there are certain residual asphalts which do not contain paraffin (i. e., obtained from asphaltic petroleums) and conversely, many native asphalts yield small percentages of paraffins upon being destructively distilled.

(12) The saturated hydrocarbons will exceed 25 per cent in the case of residual and blown asphalts, whereas they will be less than 25 per cent in the cases of native asphalts.[144] With asphaltites the saturated hydrocarbons amount to less than 10 per cent.

(13) A greater percentage of sulfonation residue is derived from residual and blown asphalts than from the various pitches.

(14) A negative diazo reaction, which distinguishes residual asphalts from pitches derived from wood, peat, lignite, coal, shale and bones.

(15) The absence of the anthraquinone reaction, which distinguishes residual asphalts from the various pitches derived from coal.

(16) By the percentage of free asphaltous acids (Test 38*a*) which run below 2 per cent in residual and blown petroleum asphalts and above 2 per cent in the native asphalts. Similarly, the acid value (Test 37*a*) of residual asphalts is usually less than 1, whereas in the case of native asphalts it is higher than 2 and may run as high as 15. Marcusson[145] reports that the saponification value (Test 37*d*) of residual asphalts is usually less than 15, and in the case of native asphalts is greater than 25, and may run as high as 36.5.

(17) In the case of natural asphalts, the "oily constituents" (Test 38*e*) are fluid at 20° C., whereas in the case of petroleum asphalts (blown or residual) they are non-fluid, having the consistency of vaseline.

There has been much discussion whether or not it is possible to distinguish between petroleum asphalts and native asphalts.[146] Various methods have been proposed for the purpose, but the only ones which seem to give dependable results are the percentages of saturated hydrocarbons (Test 34*a*), free asphaltous acids (Test 38*a*), acid value (Test 37*a*) and the saponification value (Test 37*d*), referred to in items (12) and (16) above.

At the present time it is impossible to distinguish blown asphalts from combinations of asphaltites with residual oils or soft residual asphalts, since their respective properties are very much alike.

Residual asphalts obtained from California petroleum are customarily designated by letters to differentiate the different grades. The so-called "A" grade is extremely hard and brittle, grinds to a non-adherent powder between the teeth, and ranges in penetration between 1 and 5 at 77° F. (No. 2 needle, 100 grams, 5 seconds.) "B" grade is quite hard and brittle, grinds to an adherent powder between the teeth, and ranges in penetration between 3 and 15 at 77° F. "C" grade chews with difficulty and ranges in penetration between 10 and 25 at 77° F. "D" grade chews readily without sticking to the teeth, and ranges in penetration from 25 to 75 at 77° F. "E" grade sticks to the teeth on chewing and shows a penetration greater than 75 at 77° F. "F" and "G" grades are in reality residual oils of high and low viscosities respectively. When carefully prepared, "B" grade does not contain more than 2 per

cent of non-mineral matter insoluble in carbon disulphide, and the softer grades correspondingly less.[147]

Figure 132 shows the hardness, tensile strength (multiplied by 10) and ductility curves of a typical sample of "D" grade California residual asphalt fusing at 124° F. (K. and S. method).

Table XLI includes the results obtained by the author, and Table XLII figures reported by J. Manheimer,[148] on representative specimens of residual asphalts.

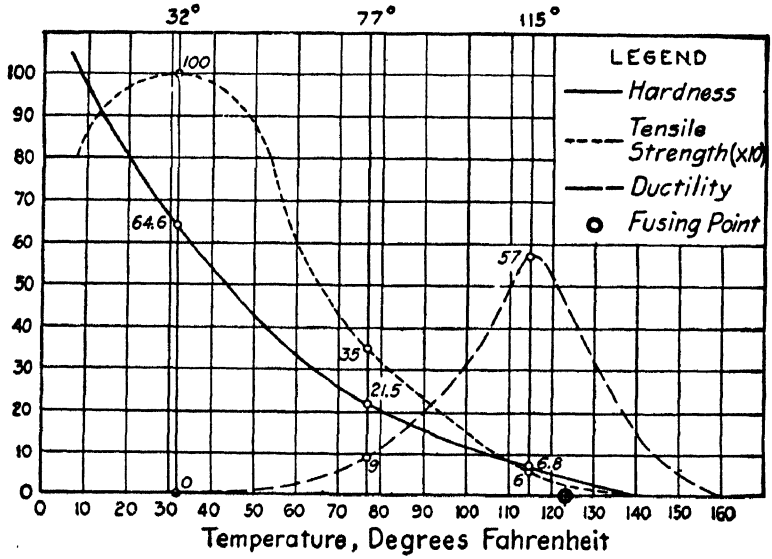


FIG. 132.—Physical Characteristics of D-Grade California Asphalt.

The weather-resisting property of residual asphalts varies, depending upon the following circumstances:

(1) The crude petroleum from which they were derived. Other things being equal, asphaltic petroleum produces the most weather-resisting residues, semi-asphaltic petroleum comes next, and non-asphaltic petroleum produces residuals having the least weather resistance.

(2) The care with which the distillation has been performed. If the residue is badly decomposed or "cracked," as evidenced by the presence of free carbon or carbenes, its weather-resisting properties will suffer in proportion.

(3) The extent to which the distillation has been carried. Soft grades of residual asphalt carrying a large percentage of oily constituents (Test 38*e*) will stand the weather better than those from which the oily constituents have been removed by driving the distillation to a point where a hard and brittle residual asphalt remains.

TABLE XLII—Characteristics of Typical Residual Asphalts

No.	Test	Mexican (Asphaltic Petroleum)			Venezuelan (Asphaltic Petroleum)			Rumanian (Non-asphaltic and Semi-asphaltic Petroleum)		German (Non-asphaltic and Semi-asphaltic Petroleum)				Polish (Non-asphaltic Petroleum)	
	Physical Characteristics:														
7	Specific gravity at 77° F	1.030	1.043	1.0575	1.012	1.035	1.042	1.014	1.031	1.026	1.065	1.066	1 082	1.038	1 050
	Mechanical Tests:														
9*b*	Penetration at 77° F	194	25	13	189	13	7	146	19 5	143	162	10	5	92.5	5 5
10*a*	Ductility at 77° F. cm	>150	5 5	17	>150	15	3 5	>150	6 5	>150	8	4	0	91	0
	Thermal Tests:														
13	Fraas breaking-point, ° F	−12	+17½	+26½	−9½	+34	+46½	+16	+32	+5	+9	+37½	+53½	+16	+57
15*a*	Fusing point (K. & S. meth.), ° F	80½	156	140	79	134	155½	88	135½	88	141	149	174	95	151
15*b*	Fusing point (R. & B. meth.), ° F	104	187	163½	101	161½	184	106	158	105	151	172½	202	112	172½
15*k*	Ubbelohde drop-point, ° F	129	211	187	123	187	211	122	172½	127½	199½	191	221	131	197
16	Volatile 325° F./5 hrs (A.S.T.M. meth.), per cent	0.287	0 033	0.0	0 022	0.00	0.004	0 168	0.113	0 0	0 0	0 0	0 0	0 02	0.01
9*b*	Residue—Penetration at 77° F	165	23.5	13	174	12	6 5	118	18	118	143	8	3.5	88	5
10*a*	Residue—Ductility at 77° F. cm	>150	4 5	10.0	>150	8 5	.....	150	5	>150	7.8	4	0	88	0
15*a*	Residue—F. P. (K. & S. meth.), ° F	83	158	140	80½	136½	158	91½	138	91½	134½	152½	177	100½	157
15*b*	Residue—F. P. (R. & B. meth.), ° F	107½	188½	161	103	163½	187	109½	160	107½	153½	177	206½	118½	179
15*k*	Residue—Ubbelohde drop-point, ° F	133	214	187	127½	190½	217½	123	179½	130	199½	195	224	133	200
16	Volatile 325° F./5 hrs. (D.I.N. meth.), per cent	1 34	0 344	0 0	0 09	0.04	0 03	0.506	0.372	0 072	0 028	0.031	0 030	0 03	0 07
9*b*	Residue—Penetration at 77° F	100	18.5	9	132	8	5.5	66	15	96	120	7	3	56	3.5
10*a*	Residue—Ductility at 77° F. cm	>150	3.5	6 5	>150	6	.....	127	4	>150	6	3	0	20	0
15*a*	Residue—F. P. (K. & S. meth.), ° F	93	169	138	89	145½	167	104	151	93	143½	156	183	109½	161
15*b*	Residue—F. P. (R. & B. meth.), ° F	118½	200½	163½	107½	167	190½	118½	172½	112	158	183	215½	127½	182½
15*k*	Residue—Ubbelohde drop-point, ° F	140	226½	188½	129	196	221	134½	190½	136½	215½	203	232	138	205
17*b*	Flash-point (open cup), ° F	450	480	622½	550½	658½	669	516	574	642	662	649½	658½	566½	674½
18	Burning-point, ° F	525	550½	694½	636	728½	722	700	700	716	720	720	722	664	752
	Solubility Tests:														
21	Soluble in carbon disulfide, per cent	99.88	99 87	99.77	99 71	99.77	99 78	98.73	98.35	98.26	98 33	96 45	95.90	97.67	99.58
	Non-min. matter insoluble, per cent	0 00	0 00	0 00	0 00	0 00	0 00	0.64	1.20	0 50	1 50	1 80	2.50	0 90	0.38
	Mineral ash, per cent	0 12	0 13	0 23	0.29	0 23	0 22	0.63	0 45	1 24	0 17	1 75	1 60	1.43	0.04
	Total, per cent	100 00	100 00	100.00	100.00	100 00	100 00	100 00	100 00	100 00	100 00	100 00	100.00	100.00	100.00
	Chemical Tests:														
28	Sulfur, per cent	6.0	6 6	6.5	3 09	3 35	3 30	0.48	0.55	1.60	1.00	1 60	1 65	0.71	0.71
33	Solid paraffins:														
	Alcohol-ether and fuller's earth, per cent	0 99	0 83	0.97	2 60	2.40	2 20	6.60	4 10	5.25	22.0	4 10	3 40	29.3	6.53
	Alcohol-ether and H_2SO_4, per cent	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	29 5	6.45
	Alcohol-ether (D. I. N. meth.), per cent	0 80	0 75	1 26	2.10	1.75	1.65	5.40	3 50	3.45	14 0	2 60	2 30	10 15	4.83
37*a*	Acid value	0 6	5 3	0 26	0.45	0 07	0 28	0 51	0 27	0.45	0.29	0 26	0 24	0.61	0.02
38	Soft asphaltic constits. (by diff.), per cent	20.8	7 7	33.6	13.10	19 9	22.6	3 45	8 55	12.22	15 40	7.80	7 00	7.03	3.40
38*c*	Asphaltenes, per cent	16.8	33 7	19.9	11.48	18 2	21.0	17 55	23 35	12 20	12 40	25 50	30 50	17 17	24.00
38*d*	Asphaltic resins, per cent	22 2	27 6	17.8	29 32	24.9	22.9	34 00	33 40	28.08	27.20	35.10	34 50	24 80	41.00
38*e*	Oily constituents, per cent	40 2	31 0	28.7	46 10	37.0	33.5	45 00	34 70	47 50	45 00	31 60	28 00	51.00	31.60
	Total, per cent	100.0	100.0	100 0	100.00	100 0	100 0	100 00	100 00	100 00	100 00	100 00	100 00	100.00	100.00
33	Solid paraffins in ditto, per cent	2.02	2.68	3 38	4 55	4 74	4.92	14 70	11 80	11 05	48 8	13 0	12.1	57.5	20.7

In general, residual asphalts of the highest quality are inferior in weather-resisting properties to native asphalts, blown asphalts, wurtzilite asphalts and fatty-acid pitches, upon comparing respective products of the same fusing-point and volatility. They are superior, however, to corresponding sludge asphalts and pitches derived from rosin, wood, peat, lignite, coal and bones.

SLUDGE ASPHALTS

Sludge asphalts are produced in much smaller quantities than residual or blown asphalts. They are derived from the purification of various petroleum distillates by means of sulfuric acid. The so-called "sludge asphalt" is present in the sulfuric acid sludge, which on cooling forms a black viscous mass. No asphalts are obtained from the caustic soda washings in the case of petroleum refining. The alkali is merely used to neutralize the sulfuric acid retained mechanically by the oil, thus differing from its action in the refining of peat and lignite tars.

The acid sludges obtained from the purification of naphtha, kerosene and lubricating oil are combined and digested with water, air and steam in a lead-lined receptacle, whereupon the lighter oily constituents rise to the surface, the acid settles to the bottom, and the tarry matters form an intermediate layer known as "acid tar" when it is soft, and "acid coke" when it is solid at ordinary temperatures. The lighter oils are withdrawn and the boiling continued until all the acid separates. The residuum is then washed with water, and heated by a spray of superheated steam until it is converted into sludge asphalt. The further the distillation is continued, the greater will be the fusing-point and hardness of the sludge asphalt. The recovered oil is known as the "acid oil distillate." The dilute sulfuric acid (specific gravity of 30° to 50° Bé.) is concentrated, first in leaden pans, and finally in an iron still until it attains a gravity of 66° Bé., when it is used over again.

Another process consists in treating the acid sludge with concentrated sulfuric acid and live steam until the mass separates into two layers, the bottom one consisting of concentrated sulfuric acid which is used over again, and the acid sludge on top. The latter is washed with hot water, which removes most of the acid, but there will still remain 3 to 15 per cent of acid mixed with the mass,

which is either heated to 250–350° C. so as to decompose the sulfuric acid into SO_2;[149] or heated in an autoclave under pressure;[150] or heated air or steam blown through the melted mass to remove the SO_2 and SO_3;[151] or the free acid may be neutralized with a residue containing naphthenic soaps;[152] or else neutralized by adding slaked lime or limestone.[153] Other methods of treating acid sludges consist in heating to not exceeding 250° C. with an equal weight of residual asphalt under agitation;[154] fluxing with 1–5 per cent of light gas-oil, emulsifying with water under agitation, breaking the emulsion by heating, treating the separated asphalt with CaO to neutralize all traces of acid, and finally distilling under vacuum to the desired consistency.[155]

The process shown diagrammatically in Table XLIII represents a method of treating acid sludges derived from heavy hydrocarbon distillates, such as lubricating oils.[156]

Acid sludges may be precipitated by salts of alkaline earths or of metals (e.g., copper or zinc), and after dehydration may be used for impregnating fabrics which are exposed continuously to moisture, preserving them from decay.[157]

Sludge asphalts test as follows:

(Test 1)	Color in mass	Black
(Test 2*a*)	Homogeneity to the eye at room temperature	Uniform
(Test 2*b*)	Homogeneity under microscope	Variable
(Test 3)	Appearance surface aged indoors one week	Bright
(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak on porcelain	Black
(Test 7)	Specific gravity at 77° F.	1.05–1.20
(Test 9*b*)	Penetration at 77° F.	150–0
(Test 9*c*)	Consistency at 77° F.	5–100
(Test 9*d*)	Susceptibility index	40–60
(Test 10)	Ductility at 77° F.	0
(Test 15*a*)	Fusing-point (K. and S. method)	80–225° F.
(Test 15*b*)	Fusing-point (R. and B. method)	100–250° F.
(Test 16)	Volatile matter 500° F. in 5 hours	2– 20 per cent
(Test 17*a*)	Flash-point	300–500° F.
(Test 19)	Fixed carbon	5– 30 per cent
(Test 21)	Solubility in carbon disulfide	95–100 per cent
	Non-mineral matter insoluble	0– 5 per cent
	Mineral matter (See NOTE)	0– 1 per cent
(Test 22)	Carbenes	0– 15 per cent
(Test 23)	Solubility in 88° petroleum naphtha	60– 95 per cent
(Test 28)	Sulfur	5– 10 per cent
(Test 30)	Oxygen	3– 7 per cent
(Test 33)	Solid paraffins	0– ½ per cent

(Test 34*a*)	Saturated hydrocarbons	Less than 10 per cent
(Test 34*b*)	Sulfonation residue	80– 95 per cent
(Test 37*e*)	Saponifiable constituents	0– 2 per cent
(Test 38*a*)	Free asphaltous acids	Less than 2 per cent
(Test 38*b*)	Asphaltous-acid anhydrides	Less than 2½ per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

NOTE. Sludge asphalts all carry traces of lead, derived from the leaden vessels in which they are produced, which is carried into solution by the sulfuric acid. The presence of lead will serve to identify sludge asphalts, and differentiate them from all other asphaltic substances. The author's investigations revealed lead varying in amounts from 0.05 to 0.25 per cent.

Sludge asphalts are characterized by the following features:

(1) Their intense black streak.

(2) Their high susceptibility index, which distinguishes them from blown asphalts.

(3) The high percentage of sulfur.

(4) The high percentage of oxygen, which distinguishes them from all other forms of asphalt and constitutes one of the most dependable tests for identifying sludge asphalts.

(5) The very small percentage of paraffin, which distinguishes them from residual asphalts obtained from mixed-base petroleum.

(6) The extremely small percentage of saturated hydrocarbons, which serves to differentiate sludge asphalts from all other asphaltic products.

(7) The comparatively large percentage of sulfonation residue, which distinguishes them from pitches.

(8) The negative diazo and anthraquinone reactions, which distinguish them from pitches derived from wood, peat, lignite, coal, shales and bones.

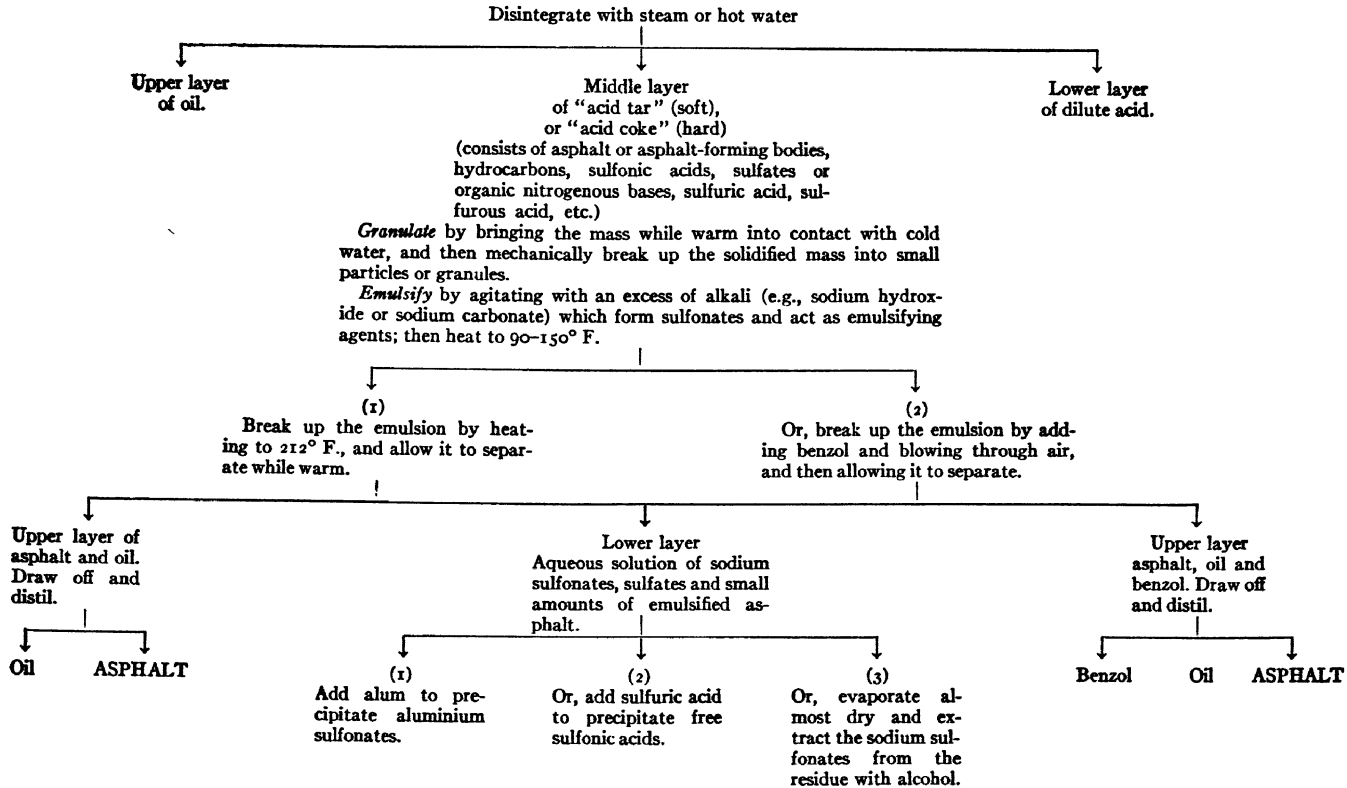
(9) The large solubility of the harder grades in 88° naphtha, which distinguishes them from residual asphalts of the same hardness and fusing-point.

Julius Marcusson [158] states that sludge asphalts may be recognized by the presence of sulfo-oxonium compounds, which on treatment with 88° petroleum naphtha will separate in the same manner as asphaltenes. They may be distinguished from the latter, however, by dissolving in pyridine and adding water. Asphaltenes will separate out, but oxonium compounds will remain in solution. They may be precipitated by adding mineral acids, ferric chloride, silver nitrate, etc. They are not affected by alkalies, but are decomposed by hydrochloric acid with the separation of sulfuric acid. A specimen of sludge asphalt examined by Julius Marcusson was found to contain 17 per cent oxonium derivatives.

TABLE XLIII

Acid Sludge

(Derived from heavy distillates, e.g., paraffin oils or lubricating oil)



Attempts have been made to blow sludge asphalts, either with air alone or by blowing in the presence of finely ground CaO.[159] However, the blowing process serves largely to harden the sludge asphalt and increase its brittleness, unless it has first been fluxed with vegetable oils.[160] Heating with $NaClO_3$ converts sludge asphalts into resinous products suitable for making molded articles.[161] Sludge asphalts flux completely with gas-works coal tar, with which it may be distilled.[162]

Typical samples of sludge asphalt examined by the author gave the results included in Table XLIV.

TABLE XLIV

CHARACTERISTICS OF TYPICAL SLUDGE ASPHALTS

No.	Test	From Various Sources, Mostly Mixtures					
	Physical Characteristics:						
2a	Homogeneity to eye at 77° F.	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.
2b	Homogeneity under microscope	Homo.	Homo.	Homo.	Homo.	Homo.	Homo.
3	Appearance surface aged 7 days	Bright	Bright	Bright	Bright	Bright	Bright
4	Fracture	.....	.....	Conch.	Conch.	Conch.	Conch.
5	Lustre	.....	.....	Bright	Bright	Bright	Bright
6	Streak	Black	Black	Black	Black	Black	Black
7	Specific gravity at 77° F.	1.057	1.052	1 068	1.090	1.076	1.155
	Mechanical Tests:						
9b	Penetration at 115° F	Too soft	Too soft	Too soft	Too soft	8	5
	Penetration at 77° F.	160	95	28	20	2	0
	Penetration at 32° F.	25	12	7	3	0	0
9c	Consistency at 115° F.	0.0	0 0	0 0	0 0	43.1	50.7
	Consistency at 77° F.	3.4	7.1	17.5	19.9	87.9	95 2
	Consistency at 32° F.	42.2	58 8	71 8	85 4	> 100	> 100
9d	Susceptibility index	49.7	59.5	61.8	53 4	> 50	> 50
10b	Ductility in cm. at 115° F.	7	15	45	3.5	0	0
	Ductility in cm. at 77° F.	38	82	18	0.75	0	0
	Ductility in cm. at 32° F.	1.5	0	0	0	0	0
11	Tensile strength in kg. at 115° F.	0 0	0.1	0.35	1.3	3.0	3 5
	Tensile strength in kg. at 77° F.	0 4	1.4	3 85	5 4	7.5	8 2
	Tensile strength in kg. at 32° F.	10.5	4.5	3.0	8 0	6 0	5 5
	Thermal Tests:						
15a	Fusing-point, deg. F. (K. and S. method)	85	99	116	160	203	210
15b	Fusing-point, deg. F.(R. and B. method)	101	113	134	178	225	230
16	Volatile, 500° F. in 5 hrs., per cent.	3 0	2.2	7.9	2.5	4.8	5.0
17a	Flash-point, deg. F.	475	500	430	480	482	486
19	Fixed carbon, per cent.	8.8	12.3	16.1	22.0	30.2	25.4
	Solubility Tests:						
21	Soluble in carbon disulfide	98.98	99 72	99.51	99.23	99.50	97.50
	Non-mineral matter insoluble	0.90	0 20	0 38	0.72	0.40	2.43
	Mineral matter	0.12	0.08	0 11	0.05	0.10	0 07
22	Carbenes	0.0	0.2	2 3	1 0	1.2	13.2
23	Soluble in 88° petroleum naphtha	93.2	94 0	78 7	74.6	66.8	62.2
	Chemical Tests:						
28	Sulfur	8.7	7.8	5.4	7.5	6.2	8.3
33	Solid paraffins	0.5	0 4	0.2	0.25	0.1	0.2
34a	Saturated hydrocarbons	9.0	8.9	7.2	5.5	4.8	3.9
34b	Sulfonation residue	82.1	88.0	.....	.....	.....	93.2

Sludge asphalts do not withstand the action of the weather as well as native asphalts, blown asphalts, residual asphalts, wurtzilite asphalt or fatty-acid pitch, comparing respective products of the same fusing-point and volatility, or of the same hardness and volatility. They are substantially equal in weather-resistance to the corresponding grades of pitch derived from coal and bones, and are superior to those derived from rosin, wood, peat and lignite. In practice, they are usually fluxed with other forms of petroleum asphalt, rather than marketed in their pure state.

A product has been marketed in the United States consisting of a mixture of sludge asphalt and pressure tar, which is decidedly inferior in weather-resisting properties. In some cases the sludge asphalt is mixed with other grades of petroleum asphalt, but with little benefit to the latter. It is claimed that the ductility of residual as well as blown asphalts at low temperatures may be improved by adding acid sludge to the topped oil prior to the distilling or blowing operation.[168]

CHAPTER XXI

PARAFFIN WAX, WAX TAILINGS AND RESINS

PARAFFIN WAX

The commercial sources of pyrogenous paraffin wax are:

(1) Peat tar;
(2) Lignite tar;
(3) Shale tar;
(4) Paraffin-bearing petroleums.

The peat-distilling industry is comparatively unimportant and does not form a factor in the production of paraffin. The lignite industry has only attained commercial importance in Germany, and the shale industry in Scotland. The treatment of paraffin-bearing petroleums for the recovery of paraffin is important the world around. The methods for recovering paraffin wax from lignite tar, shale tar and petroleum are substantially the same. In the case of lignite tar, the paraffin wax is obtained from the distillate fractioned after the crude oil, known as the "paraffinaceous mass." With shale tar the paraffin wax is obtained from the distillate known as "heavy oil," distilling after the "intermediate or gas-oil." With paraffin-bearing petroleums, the paraffin wax is obtained from the fraction known as the "paraffin distillate."

For a description of the methods of refining and purifying paraffin wax, the reader is referred elsewhere.

Paraffin wax, including commercial "refined paraffin wax" and "refined scale wax" tests as follows:

(Test 1)	Color in mass	Pure white to yellowish
(Test 2*a*)	Homogeneity to the eye at room temperature.	Uniform to slightly crystalline
(Test 2*c*)	Homogeneity when melted	Uniform and transparent
(Test 4)	Fracture	Conchoidal to hackly
(Test 5)	Lustre	Dull and "waxy"
(Test 6)	Streak	White
(Test 7)	Specific gravity at 77° F.	0.85– 0.95
(Test 9*b*)	Penetration at 77° F.	5–50

(Test 9*c*)	Consistency at 77° F.	15–80
(Test 9*d*)	Susceptibility index	> 100
(Test 10)	Ductility at 77° F.	0
(Test 15*b*)	Fusing-point (R. and B. method)	105–160° F.
(Test 15*f*)	Fusing-point (Special method)	100–150° F.
(Test 16)	Volatile at 325° F. in 5 hours	20–40 per cent
	Volatile at 500° F. in 5 hours	33–60 per cent
(Test 17*a*)	Flash-point	Comparatively low
(Test 18)	Burning-point	Comparatively low
(Test 19)	Fixed carbon	0– 2 per cent
(Test 21)	Solubility in carbon disulfide	99–100 per cent
	Non-mineral matter insoluble	Trace
	Mineral matter	Trace
(Test 22)	Carbenes	0 per cent
(Test 23)	Solubility in 88° petroleum naphtha	99–100 per cent
(Test 30)	Oxygen	Trace
(Test 33)	Solid paraffins	95–100 per cent
(Test 34*a*)	Saturated hydrocarbons	90– 99 per cent
(Test 34*b*)	Sulfonation residue	95–100 per cent
(Test 37*e*)	Saponifiable constituents	0 per cent
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Three grades of paraffin wax are specified by the U. S. Government, having fusing-points (Test 15*f*) of 130–132° F., 124–127° F. and 117–120° F. respectively. Normal "slack wax" has a fusing-point of 125° F. Various fusing-points are demanded by the trade.

Paraffin wax is remarkably resistant to the action of chemicals, but on exposure to the weather, the oily constituents soon evaporate, leaving a pulverulent and but slightly coherent mass behind. This is not due to oxidation, but merely to volatilization of the oils present in the wax.

Paraffin wax withstands the continuous action of water very well and finds a ready market for preparing waterproof papers, for manufacturing candles, for household purposes, in miners' lamps, manufacture of matches, drinking cups, bottle caps, cartridge containers, dry cells, wax figures, molds, crayons, etc.

WAX TAILINGS

This product is obtained during the dry distillation of non-asphaltic or semi-asphaltic petroleum. The residuum left in the retort at the end of the first distillation is subjected to a second process of dry distillation, whereupon the wax tailings distils over, just prior to the formation of coke. Wax tailings is sometimes

termed "still wax," although both these names are misnomers, since it contains only small quantities of paraffin wax. It consists largely of decomposition products, including chrysene, picene and anthracene, and has a decided yellow color, by which it is recognized during the process of distillation. Upon cooling it forms a very viscous semi-liquid to sticky semi-solid of a characteristic light yellow to yellowish brown color. It complies with the following tests:

(Test 1)	Color in mass	Yellow to yellowish brown
(Test 2*a*)	Homogeneity to the eye at room temperature	Uniform to very slightly granular
(Test 2*b*)	Homogeneity under microscope	Uniform to gritty (due to the crystalline constituents present)
(Test 3)	Appearance surface aged indoors	Variable
(Test 5)	Lustre	Waxy
(Test 6)	Streak	Pale yellow
(Test 7)	Specific gravity at 77° F.	1.00–1.10
(Test 9*b*)	Penetration at 77° F.	10–150
(Test 9*c*)	Consistency at 77° F.	5–20
(Test 9*d*)	Susceptibility index	20–40
(Test 10)	Ductility at 77° F.	Usually quite high
(Test 15*a*)	Fusing-point (K. and S. method)	60–100° F.
(Test 15*b*)	Fusing-point (R. and B. method)	65–110° F.
(Test 16*a*)	Volatile matter at 500° F., 5 hours	5–10 per cent
(Test 17*a*)	Flash-point	300–450° F.
(Test 19)	Fixed carbon	2–8 per cent
(Test 21)	Solubility in carbon disulfide	98–100 per cent
	Non-mineral matter insoluble	0–2 per cent
	Mineral matter	0–Trace
(Test 22)	Carbenes	0–Trace
(Test 23)	Solubility in 88° petroleum naphtha	95–100 per cent
(Test 28)	Sulfur	0–Trace
(Test 30)	Oxygen	0–2 per cent
(Test 32)	Naphthalene	Absent
(Test 33)	Solid paraffins	Tr.–5 per cent
(Test 34*a*)	Saturated hydrocarbons	40–70 per cent
(Test 34*b*)	Sulfonation residue	90–100 per cent
(Test 37*c*)	Saponifiable constituents	Trace
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	Yes

Wax tailings is an exceedingly good flux, and will thoroughly amalgamate with the various pitches, likewise with the harder asphalts and asphaltites, including even grahamite.[1] It forms a better flux than the residual oils derived from asphaltic petroleum. A very small percentage will often serve thoroughly to flux materials

which are otherwise incompatible, and at the same time increase the ductility of the mixture. Certain asphalts, although they may flux together at high temperatures, will separate partially on cooling, forming a very finely granular condition, which is particularly noticeable when the surface of the mixture is freshly disturbed, or upon drawing a small pellicle into a thread. The presence of a small percentage of wax tailings will often prevent this, and it therefore enjoys a unique position among the fluxes. Large quantities, however, should be avoided, as wax tailings is extremely susceptible to changes in temperature and lacks weather-proof properties. The presence of wax tailings will increase the solubility of asphaltic substances in petroleum distillates, and accordingly becomes useful for manufacturing certain types of bituminous paint. A representative sample of wax tailings tested by the author gave the following results:

(Test 9*b*)	Penetration at 165° F.	Too soft
	Penetration at 77° F.	100
	Penetration at 32° F.	51
(Test 9*c*)	Consistency at 115° F.	0.0
	Consistency at 77° F.	5.9
	Consistency at 32° F.	22.9
(Test 9*d*)	Susceptibility index.	25.0
(Test 10*b*)	Ductility at 115° F.	1.1
	Ductility at 77° F.	3.9
	Ductility at 32° F.	13.5
(Test 11)	Tensile strength at 115° F.	0.0
	Tensile strength at 77° F.	0.5
	Tensile strength at 32° F.	9.5
(Test 15*a*)	Fusing-point (K. and S. method)	90° F.
(Test 15*b*)	Fusing-point (R. and B. method)	98° F.
(Test 17*a*)	Flash-point	382° F.

A process has been proposed for coloring wax tailings for use as a saturant in the manufacture of saturated fabrics, felt-base floor coverings, paints, etc., which consists in distilling the wax tailings with superheated steam to a semi-solid or solid consistency and then combining with a soluble dye such as rhodamine, malachite green, etc.; or else forming a mechanical mixture with a colored mineral pigment.[2] Wax tailings may also be vulcanized by heating with sulfur.[3] The production of wax tailings is not large, and hence it is not of great importance to the asphalt industry.

PETROLEUM RESINS

These have been produced by distillation of certain crude petroleums (e.g., Miri) which are free from asphaltenes, or by the action of anhydrous aluminium chloride (about 2 per cent) on an unsaturated distillate obtained by cracking at a high temperature and low pressure, having a specific gravity at 20° C. of 0.84–0.86 and a boiling range of 23–180° C. The reaction mixture is treated with NaOH to precipitate the aluminium from the aluminium chloride polymer, whereupon the resin remains in solution and is recovered by distillation under reduced pressure, at as low a temperature as possible. The resin is a hard, amber-colored product, having a fusing-point of 230–240° F. (R. and B.), which is soluble in practically all hydrocarbon solvents, but insoluble in methanol, ethyl alcohol and acetone. It dissolves readily in drying oils, and has an acid value between 0.1 and 2. When a film is baked at 220° F. for 1 hour, it becomes insoluble in its original solvents. Petroleum resins are claimed to accelerate the drying properties of linseed and china-wood oils.[4]

ASPHALTIC RESINS

A process has recently been commercialized for producing light-colored asphaltic substances falling within the chemical classification of "asphaltic resins" (see Test 38*d*), by removing the asphaltenes in accordance with the following procedure: (1) precipitating the asphaltenes with an agent, such as gasoline, pentane, etc.; (2) treating with chemicals, such as sulfuric acid or sulfur trioxide, which converts the asphaltenes into an insoluble modification; and (3) treating with decolorizing agents, such as fuller's earth or carbon, with or without diluting agents. An example consists in dissolving residual asphalt derived from Java petroleum in twice its weight of light petroleum naphtha, then adding 2 per cent by weight of sulfuric acid, removing the acid tar and enveloping substances by settling, adding 3 per cent by weight of bleaching earth, and finally filtering. The naphtha is thereupon removed from the filtrate by evaporation and the resulting light-colored asphalt steam-distilled to the required consistency.[5] Its physical characteristics are quite similar to wax tailings. It is characterized by a specific

gravity at 77° F. of 1.02–1.04, fusing-point (R. and B.) of 100–130° F., high ductility, brittleness at low temperatures, great susceptibility to temperature changes, low viscosity when liquefied, and high adhesiveness. It may be fluxed with low-grade asphalts to improve the qualities of the latter.[6] The product has been recommended for use in preparing aqueous dispersions suitable for paints in combination with colored pigments, for impregnating fabrics, and for other purposes where the light color will prove an advantage. Another process consists in precipitating the asphaltenes from a solution of the asphalt in gasoline or petroleum naphtha, by the addition of colloidal silicic acid, whereupon the solution is decanted and evaporated;[7] also a process of distilling pressure tar with steam under vacuum (with or without the addition of $AlCl_3$) collecting a distillate having a specific gravity of 1.084–1.126, a fusing-point (R. and B.) of 93–150° F., and a ductility at 77° F. of over 100 cm., said product being a substantially solid resin at ordinary temperatures.[8] This product has been recommended for the manufacture of pigmented paints and lacquers.[9]

CHAPTER XXII

WURTZILITE ASPHALT

Wurtzilite asphalt or wurtzilite pitch, marketed under the name of "kapak," is produced by cracking or depolymerizing wurtzilite. It is similar to the latter in its physical characteristics with the exception of:

(1) The hardness, which is very much reduced.
(2) Its fusibility. Treated wurtzilite is fusible whereas crude wurtzilite is not.
(3) Its solubility. Treated wurtzilite is readily soluble in carbon disulfide, and moderately so in 88° petroleum naphtha, whereas the crude product is practically insoluble in both.

The process consists in heating the wurtzilite in a closed vessel or still to a temperature of 500 to 580° F., under more or less pressure. Vapors are evolved during the process that are condensed and returned to the still, and in turn attack the wurtzilite, first reducing it to a plastic mass, which after heating is converted into a fusible substance. If the vapor pressure becomes too great, some is allowed to escape. The process which takes place is virtually a "depolymerization." [1]

Another process consists in grinding together wurtzilite and gilsonite and then heating together for some time at 350° C. until readily fusible, whereupon the mass is fluxed with petroleum asphalt.[2]

In practice, the wurtzilite is first run through a crusher to break up any coarse lumps, and then fed into a horizontal cylindrical still through two charging hoppers, one at either end, provided with tightly fitting covers which are fastened into place before the fires are started. The bottom of the still is protected by a fire-brick arch, and the products of combustion after passing underneath the arch, are returned in three fire-flues running through the still (one 10 in. in diameter and two 6 in.), and thence back again in the space

surrounding the still, above the arch. The vapors generated from the wurtzilite pass upward through two pipes joined to the top of the still, near the ends, and connected with a single water-cooled coil, which condenses the vapors and returns most of the condensate to the still. Not all the condensate is returned, however, for practice has demonstrated that the optimum results are obtained if 2 to 5 per cent of the distillate (based on the weight of wurtzilite charged into the still) is drawn off. This may be used as fuel under the still.

The longer the wurtzilite is heated in its process of manufacture the greater will be the quantity of oils produced, which act in the same manner as a flux, and hence the fusing-point and hardness of the resultant product will be lower.

It takes six to eight hours to raise the temperature of the charge to 400° F., then four to six hours to reach the maximum temperature (580° F.), which is maintained from twenty-four to thirty-six hours. The contents are then allowed to cool to 450° F., and finally drawn off through a valve at the bottom.

According to the author's investigations, Nova Scotia albertite is also amenable to this process, although up to the present time it has not been thus treated commercially.

The wurtzilite asphalts are marketed under various arbitrary numbers ranking from "0" to "16," each of which is recommended for a specific purpose, including the manufacture of paints and varnish, insulators, for manufacturing insulated wire, for weatherproofing conduits and cables, as a filler for mechanical and hard rubber compounds cured by the press or open steam method, for coating prepared roofings, for manufacturing carriage drills and similar compositions applied by the calendar process and cured by the dry-heat method, etc. Certain of these compounds represent mixtures of wurtzilite asphalt and gilsonite, with or without the addition of asphaltic fluxes (e. g., residual oil), and vegetable oils (e. g., linseed oil, palm oil, etc.).

Wurtzilite asphalt complies in general with the following characteristics:

(Test 1)	Color in mass	Black
(Test 2*a*)	Homogeneity to the eye at room temperature	Uniform
(Test 2*b*)	Homogeneity under microscope	Uniform
(Test 3)	Appearance surface aged indoors one week	Very bright

(Test 4)	Fracture	Conchoidal
(Test 5)	Lustre	Bright
(Test 6)	Streak	Brown to black
(Test 7)	Specific gravity at 77° F.	1.04–1.07
(Test 9*b*)	Penetration at 115° F.	15–25
	Penetration at 77° F.	5–20
	Penetration at 32° F.	0–15
(Test 9*c*)	Consistency at 115° F.	10–25
	Consistency at 77° F.	20–50
	Consistency at 32° F.	50–120
(Test 9*d*)	Susceptibility index	30–40
(Test 10*b*)	Ductility at 115° F.	1–5
	Ductility at 77° F.	0–1
	Ductility at 32° F.	0
(Test 11)	Tensile strength at 115° F.	1–4
	Tensile strength at 77° F.	5–10
	Tensile strength at 32° F.	8–15
(Test 15*a*)	Fusing-point (K. and S. method)	150–300° F.
(Test 15*b*)	Fusing-point (R. and B. method)	170–325° F.
(Test 16*a*)	Volatile at 500° F., 5 hours	Less than 5 per cent
(Test 17*a*)	Flash-point	450–600° F.
(Test 19)	Fixed carbon	5–25 per cent
(Test 21)	Solubility in carbon disulfide	98–100 per cent
	Non-mineral matter insoluble	0–½ per cent
	Mineral matter	Tr.–2 per cent
(Test 22)	Carbenes	0–2 per cent
(Test 23)	Solubility in 88° petroleum naphtha	50–80 per cent
(Test 28)	Sulfur	4–6 per cent
(Test 30)	Oxygen	0–2 per cent
(Test 33)	Solid paraffins	0–Trace
(Test 34*a*)	Saturated hydrocarbons	5–12 per cent
(Test 34*b*)	Sulfonation residue	90–95 per cent
(Test 37*e*)	Saponifiable constituents	Trace
(Test 39)	Diazo reaction	No
(Test 40)	Anthraquinone reaction	No

Specimens of the unfluxed wurtzilite asphalt examined by the author tested as follows:

		No. 0	No. 1	No. 6	No. 16
(Test 9*b*)	Penetration at 115° F.	16	19	23	34
	Penetration at 77° F.	6	7	9	12
	Penetration at 32° F.	0	1	4	7
(Test 9*c*)	Consistency at 115° F.	22.9	18.5	15.0	11.7
	Consistency at 77° F.	45.6	41.8	38.5	32.4
	Consistency at 32° F.	110.0	92.2	80.6	71.1
(Test 9*d*)	Susceptibility index	32.4	33.4	32.8	31.2
(Test 15*a*)	Fusing-point (K. and S. method)	269	220	200	190
(Test 10*b*)	Ductility at 115° F.	2	3	3	4
	Ductility at 77° F.	0	0	0	½
	Ductility at 32° F.	0	0	0	0

These figures indicate that the extent of softening and lowering of the fusing-point is dependent upon the extent to which the process of depolymerization has progressed. It is interesting to observe in this connection that the susceptibility index remains practically unchanged.

Wurtzilite asphalt is characterized by its low specific gravity, high fusing-point, low susceptibility index, extreme toughness and rubber-like properties (i. e., resiliency), high-tensile strength, small percentages of oxygen and non-mineral matter, large percentage of sulfonation residue, and absence of saponifiable constituents.

It is quite similar in many respects to blown asphalts (particularly in regard to its susceptibility index), but may be differentiated from these by:

(1) A greater hardness or consistency at 77° F. for any given fusing-point.
(2) A greater tensile strength for any given fusing-point.
(3) Smaller percentages of oxygen.
(4) Smaller percentages of saturated hydrocarbons.

Wurtzilite asphalt is also similar in many respects to the fatty-acid pitches, especially in its toughness (resilience) and its low susceptibility index. It is distinguished from these, however, by the following:

(1) Its solubility in 88° petroleum naphtha, which is smaller than in the case of fatty-acid pitches.
(2) The presence of sulfur, which is absent in the fatty-acid pitches.
(3) The smaller percentage of oxygen.
(4) The larger percentage of sulfonation residue.
(5) The absence of saponifiable constituents.

In other respects they are apt to test pretty much alike.

Wurtzilite asphalt shows remarkable weather-resistance and finds its greatest use in manufacturing asphalt paints, for coating prepared roofings,[3] and for coating cotton-covered wires for electrical insulation.[4] Its use is limited by the small quantity produced, and the comparatively high price at which it is marketed.

PART V

MANUFACTURED PRODUCTS AND THEIR USES

CHAPTER XXIII

COMPOUNDING OF BITUMINOUS SUBSTANCES

GENERAL CONSIDERATIONS

One of the most important questions which confronts the bitumenologist is that of blending the various substances at his disposal, to produce mixtures best adapted for the special purposes for which they are intended. This requires an intimate knowledge of the nature and behavior of the various materials, and can only be thoroughly acquired by years of experience. Two shipments of any given member of the bituminous family are likely to fluctuate widely in their physical properties and composition, even when procured from the identical source. A native bituminous substance emanating from the *same* deposit will vary, depending upon the degree of exposure and amount of metamorphosis. It has been shown that all native bituminous materials are in a constant state of transition, depending upon their age and environment. Scarcely any two deposits of native asphalt are alike in their properties or chemical composition. The same applies to petroleum, which varies in different localities and very often in wells side-by-side in the same field.

The pyrogenous bituminous materials also show a marked variation in their properties, depending upon the raw materials used in their production and the exact conditions to which they have been subjected in their process of manufacture, including the temperature, length of treatment, etc.

Bituminous materials should not therefore be compared to vegetable and animal fats or oils, which in the case of any one material runs fairly uniform in composition and physical properties.

The consistency of the available ***raw*** bituminous materials is

either fixed and definite, or it is controllable. Each native bituminous substance has a predetermined consistency—in other words, it is endowed by nature with certain fixed and definite physical properties, and over which man has no control. On the other hand, the consistency of pyrogenous bituminous substances, with three exceptions (i.e., wax tailings, tars and rosin pitch), is largely controllable, and depends upon the treatment to which they are subjected in the course of their production. In the majority of pyrogenous substances, it is a comparatively easy matter to alter their consistency at will, by regulating the duration of the process, its temperature, or some other condition. In the three exceptions noted, the consistency is predetermined, and has no definite bearing upon the variable factors of the process.

Table XLV will serve to give a general idea whether the hardness and fusibility of the various bituminous substances are definite or controllable, whether the substances are naturally soft, medium or hard at room temperature; also their approximate comparative volatility, weather-proof properties, and efficiency in fluxing.

In interpreting Table XLV, it should be distinctly noted that the "Hardness at Room Temperature," "Volatility," "Weather-proof Properties" and "Efficiency in Fluxing" are listed in a *comparative* sense, and must not therefore be regarded as a definite exposition of the characteristics of any *single* substance, without taking into consideration the other substances cited.

In addition to the bituminous substances, there are included three groups of non-bituminous substances commonly used for purposes of blending; viz., rosin, animal and vegetable oils or fats, and wool grease.

In preparing mixtures of bituminous materials, the following points should be borne in mind:

(1) Bituminous materials which give the diazo reaction (Test 39) (containing phenols), should not be mixed with bituminous substances not giving this reaction. In other words, native asphalts, asphaltites and pyrogenous asphalts should not be blended with tars or pitches (excepting fatty-acid pitch), since it has been found by experience that such mixtures, although they may melt together perfectly, are not durable or weatherproof, although such mixtures have been used successfully for sub-soil waterproofing purposes and certain types of pavements.

TABLE XLV

	Hardness and Fusibility, as Found or Produced	Penetration at 77° F. (Test 9b)	Approximate Comparative Volatility	Approximate Comparative Weatherproof Properties	Efficiency in Fluxing	Remarks
Legend...	D: definite. FC: fairly definite but controllable within narrow limits. IC: indefinite but controllable. IN: indefinite and noncontrollable.	S: soft. [>25] M: medium [15–25] H: hard [<15]	V: volatile. M: moderately volatile. N: non-volatile.	H: highly weatherproof. W: weatherproof. M: moderately weatherproof. N: non-weatherproof.	G: good. F: fair. P: poor.	
BITUMINOUS SUBSTANCES:						
BITUMENS:						
Crude Petroleums	D	S	V	N	P	Rarely used for blending.
Native mineral waxes:		M	M to N	M	P	Used for imparting wax-like properties, an unctuous feel, or to lessen the tendency towards stickiness.
Ozokerite	FC	H	N	M	P	
Montan wax	D	H	N	W to H	F to G	
Native asphalts	IN	S	V to M to N	M to W to H	G	Blend with one another, also with pyrogenous asphalts, asphaltites and fatty-acid pitches.
		M	M to N	M to W to H	G	
		H	N	W to H	G	
Asphaltites	D	H	N	H	G*	Used to harden and raise the fusing-point of asphaltic mixtures.
PYROGENOUS DISTILLATES:						
Pyrogenous waxes	IC	S	V to M	N to M	P	Same as for ozokerite and montan wax.
		M	M	M	P	
		H	M to N	M	P	
Wax tailings	D	S	N	N to M	G	Excellent flux for all bituminous substances, but not weatherproof.
Tars	D	S	V	N	G	Rarely used for fluxing.
PYROGENOUS RESIDUES:						
Pyrogenous Asphalts:						
Residual oils (from non-asphaltic crudes)	FC	S	M to N	W to H	P to F	Excellent flux for all asphaltic mixtures, asphaltites * and fatty-acid pitches.
Residual oils (from semi-asphaltic crudes)	FC	S	M to N	W to H	G	Excellent fluxes for all asphaltic materials, all asphaltites and fatty-acid pitches.
Residual oils (from asphaltic crudes)	FC	S	M to N	W to H	G	

* Excepting grahamite.

TABLE XLV (*Continued*)

Pressure tars	IC	S	V	N to M	G	
Blown petroleum asphalts	FC	S	N	W to H	G	
		M	N	W to H	G	
Residual asphalts	IC	S	N	W to H	G	
		M	N	W to H	G	
		H	N	W	G	Blend readily with asphaltic mixtures, asphaltites and fatty-acid pitches.
Sludge asphalts	IC	S	N	N to M	G	
		M	N	N to M	G	
		H	N	N to M	G	
Wurtzilite asphalt	FC	M	N	W to H	G	
		H	N	W to H	G	
Pitches:						
Wood-tar pitch	IC	S	V to M	N	G	Rarely used for fluxing.
		M	M to N	N	G	Blend readily with all pitches.
		H	N	N	G	
Rosin pitch	D	H	M to N	N	G	An excellent flux for all bituminous substances, but not weatherproof.
Peat- and lignite-tar pitch	IC	S	V to M	N to M	G	Rarely used for fluxing.
		M	M to N	M	G	Blend readily with all pitches.
		H	N	M	G	
Water- and oil-gas-tar pitch	IC	S	V to M	N to M	G	Rarely used for fluxing.
		M	M to N	M to W	G	Blend readily with all pitches.
		H	N	W	G	
Coal-tar pitches	IC	S	V to M	N to M	G	Rarely used for fluxing.
		M	M to N	W	G	Blend readily with all pitches.
		H	N	W	G	
Fatty-acid pitch	IC	S	N	W to H	F to G	Blend readily with all bituminous substances,* also weatherproof.
		M	N	W to H	F to G	
		H	N	W to H	F to G	
Bone-tar pitch	IC	S	V to M	N to M	G	Rarely used for fluxing.
		M	M to N	M	G	Blend readily with all pitches.
		H	N	W	G	
NON-BITUMINOUS SUBSTANCES:						
Rosin	D	H	M to N	N	G	Excellent flux for all bituminous substances but not weatherproof.
Animal or vegetable oils and fats	D	S	N	H	G	Excellent fluxes for all bituminous substances and highly weatherproof
Wool grease	D	S	N	H	F to G	Fluxes readily with most bituminous substances* and highly weatherproof.

* Excepting grahamite.

(2) Native and pyrogenous waxes will not remain permanently blended with other bituminous materials, but will crystallize at low temperatures, gradually separating from solid mixtures on standing. This manifests itself by the wax "sweating" from the surface. In certain cases this feature is desirable, since the admixture of a small percentage of wax imparts wax-like properties to the entire composition.

(3) Grahamite does not flux with native or pyrogenous waxes, residual oils derived from non-asphaltic petroleum, fatty-acid pitch or wool grease.

(4) If the bituminous material contains a percentage of "non-mineral matter insoluble in carbon disulfide," the act of fluxing with other bituminous substances, rosin, animal or vegetable oils and fats will only dilute this ingredient, without eliminating it. The calculated proportion will still be present in the mixture.

(5) The percentage of carbenes may be reduced by fluxing or blending, as the carbenes themselves are fluxed by other bituminous substances, rosin, animal or vegetable oils and fats.

(6) The thoroughness with which bituminous materials blend may be ascertained by finding the fusing-point of the components, and comparing the calculated fusing-point of the mixture with its actual fusing-point. If the actual fusing-point is equal to, or greater, than the calculated fusing-point, then the blending has been thorough. On the other hand, if the actual fusing-point is less than the calculated fusing-point, then the components do not amalgamate thoroughly.

(7) The microscopic test may also be used to good advantage to ascertain the thoroughness with which the components blend. A separation of particles in the mixture is evidence that perfect amalgamation does not occur.

Where the hardness and fusibility of the bituminous substance are "controllable," it is often a simple matter to continue the distillation, blowing, depolymerization (in the case of wurtzilite asphalt), etc., until a product is obtained having the desired physical characteristics. Where conditions permit, it is more convenient and economical to turn out a product of *exactly* the proper grade, than to flux or harden it *afterwards*. It is accordingly customary to market residual asphalts of exactly the right hardness and fusing-point for paving purposes, etc. Blown asphalts are similarly marketed in various grades, having different fusing-points (and hardnesses), so that the manufacturer may pick out one best adapted to his particular purpose. Coal tar is likewise distilled to a predeter-

mined extent to obtain pitches suitable for use as such in connection with waterproofing, roofing work, road purposes or briquette making, whichever the case may be.

Binary Mixtures. It is not always possible to use a single bituminous material, since it is sometimes found that the *exact* characteristics required are lacking, and can only be obtained by blending together two or more substances in suitable proportions. The simplest mixtures, containing two constituents, are known as "binary mixtures." In this case it is often possible to predict with a fair degree of accuracy what the characteristics of the mixture will be. That this is not always a simple matter is indicated by the following figures. The same blown asphalt was used in both cases, whereas the steam-refined asphalts A and B differed slightly:[1]

TABLE XLVI

Per Cent of Blown Asphalt Combined with:	Penetration at 77° F. (Test 9*b*)	R. and B. Fusing-Point deg. F. (Test 15*b*)	Ductility at 77° F. (Test 10*a*)
Steam-refined Asphalt "A":			
0	30	131	115
10	34	130	115
20	49	129	115
30	65	124	115
40	73	126	115
45	77	127	115
50	67	134	57.5
60	60	145	12.5
100	37	270	1.5
Steam-refined Asphalt "B":			
0	30	132	115
10	35	132	115
20	44	129	115
30	54	136	78
40	56	143	19.5
50	57	151	9.5
70	50	173	4.25
100	37	270	1.5

The purpose of preparing a *mixture* is to soften the substance and lower its fusing-point, harden the substance and raise its fusing-point, render the mixture less susceptible to temperature changes, effect a more perfect union or blending of the constituents, improve its weatherproof properties, increase the tensile strength, render the

mixture wax-like, or unctuous to the feel, lessen the tendency towards stickiness, etc.

Softening the Substance and Lowering Its Fusing-point. This process is ordinarily known as "fluxing." When the bituminous material, as it occurs naturally or results from a manufacturing process is too hard in consistency or fuses at too high a temperature, it is customary to mix it with a softer substance, termed a "flux," to impart the necessary characteristics. The fluxes may be classified in three groups as follows:

Group I For Softening Asphaltic Materials and Asphaltites	Group II For Softening Pitches	Group III Used Indiscriminately for Softening Asphaltic Materials, Asphaltites and Pitches
Soft native asphalt	Medium wood tar pitch	Wax tailings
Residual oils	Medium peat-and lignite-tar pitches	Soft and medium fatty-acid pitches*
Pressure tars	Medium water-gas-tar pitch	Animal and vegetable oils and fats
Soft blown asphalt	Medium oil-gas-tar pitch	Wool grease*
Soft residual asphalt	Medium coal-tar pitch	
Soft sludge asphalt	Medium bone-tar pitch	

* Not suitable for fluxing grahamite.

The fluxes listed in Group II should not be used for softening asphaltic materials and asphaltites, or those in Group I for softening pitches (excepting rosin- and fatty-acid pitches), for reasons already explained. The fluxes listed in Group III will answer satisfactorily for softening asphaltic products, asphaltites and pitches, without injuring their weather-resisting qualities.

Of Group I fluxes: residual oils, soft blown asphalt and soft residual asphalt are ordinarily used, on account of their weather-resisting properties, their efficiency in fluxing, the absence of volatile constituents and their comparative cheapness.

Of Group II fluxes: water-gas-tar pitch and oil-gas-tar pitch are ordinarily used for preparing "cut-back" coal-tar pitches for use as dust-laying oils, road surfacings, etc.

Of Group III fluxes: animal and vegetable oils or fats are most generally employed, owing to their abundance and uniformity. They are only used in *special cases* (e.g., manufacturing certain bituminous lacquers, varnishes and japans, rubber substitutes, coating compositions for high-grade prepared roofings, etc.), in view of their comparatively high price, although they are without question the fluxes *par excellence* for bituminous materials.

Augmenting the Adhesive Properties of the Substance. Bituminous substances may have their adhesiveness enhanced by fluxing with a small percentage (e.g., 0.1 to 2 per cent) of the following materials: an aromatic or aliphatic alcohol (e.g., fusel oil);[2] cyclohexanone, $CO.Et.C_5H_{11}$, $Bu_2C_2O_4$, etc.;[3] naphthalene or hydronaphthalene dissolved in alcohols or ketones;[4] naphthenic acids;[5] solid fatty acids (e.g., stearic), resin acids or oxidized paraffin wax;[6] polymerized vinyl chloride;[7] crude phenols;[8] "rubber oil" obtained in the destructive distillation of rubber;[9] etc.

Increasing the Fluidity of the Substance When Melted. Another group of materials is of interest in connection with saturants. They have been termed "introfiers" (i.e., impregnation accelerators),[10] and when added to an impregnating material in relatively small quantities will result in a marked change in its fluidity and wetting properties towards the material being impregnated, increasing the speed of penetration. This group of materials includes substances of the type of naphthalene (e.g., naphthalene, naphthols, naphthylamines, hydronaphthalenes, chloronaphthalenes, etc.); pyridine bases (e.g., pyridine, picolines, lutidines, quinoline, quinaldine, acridine, or similar compounds);[11] nitrogenous distillation products (e.g., dipped oil, bone oil, shale oil, etc.);[12] also of the type of diphenyl (e.g., diphenyl, diphenylmethane, dibenzyl, phenyl ether, fluorene, etc.); also of the type of triphenyl (e.g., triphenylmethane, triphenylchloromethane, triphenylphosphate, etc.); likewise paracumarone ("cumar") resin;[13] etc.

Effecting a More Perfect Union or Blending of the Constituents. At times the components of a bituminous mixture do not amalgamate thoroughly. This is detected by a lack of homogeneity (Test 2), i.e., by distributing the surface and observing whether it becomes duller, or else by drawing out a pellet into a thin thread, and noting whether any dulling occurs. Certain fluxes when combined with the mixture, often in a small proportion, serve to overcome this tendency, and result in a more complete amalgamation of the components. Such fluxes in the approximate order of their efficiency are as follows:

Rosin	Animal or vegetable oils and fats
Rosin pitch	Wax tailings

It will be understood that these fluxes do not influence the per-

centage of non-mineral matter insoluble in carbon disulfide (free carbon), or any dullness due to this constituent.

In general it may be stated that petroleum asphalts and coal-tar pitch do not readily amalgamate except in restricted proportions.[14]

Gilsonite has been recommended as an addition to asphalts for the purpose of "correcting" heterogeneity.[15] Waxy substances (e.g., paraffin wax) are better tolerated by some asphalts than by others, but may be combined in nearly all proportions with gilsonite selects, and without danger of subsequent separation, which fact is taken advantage of in the insulated-wire industry.

A greater percentage of natural asphalts (e.g., Trinidad asphalt) may be blended with coal tar and coal-tar pitch without coagulating the free carbon present in the latter, than proves to be the case with petroleum asphalts.[16]

Asphalts having the highest surface-tension flux best with tars,[17] and homogeneous mixtures are favored through the use of *soft* tars and *hard* asphalts.[18] The addition of the following substances will promote the fluxing of coal-tar pitches and petroleum asphalts: rosin or wax tailings;[19] anthracene oil;[20] Dippel's oil or shale oil, and nitrogenous bases;[21] organic compounds of heavy metals (e.g., lead oleate or lead resinate);[22] certain forms of colloidal mineral matter (e.g., Al_2SlO_3 or Al_2O_3).

Coal-tar pitches will flux in all proportions with vegetable oils (plain or vulcanized) and fatty-acid pitches,[23] and coke-oven tar will blend in all proportions with pressure tars derived from petroleum.[24]

Mixtures of coal tar and asphalt are favored for road construction in Europe and Great Britain, it being claimed that such mixtures have the following advantages over the use of tar alone:

(1) Have increased viscosities at elevated temperatures.
(2) Are less susceptible to temperature changes.
(3) Possess greater adhesive qualities.
(4) Have greater weather-resistance.

Up to 20 or 25 per cent petroleum asphalt or coal-tar pitch respectively may be incorporated in the mixture and form a homogeneous composition. If more than 25 per cent asphalt is fluxed with coal-tar pitch, or conversely, if more than 25 per cent of coal-tar pitch is fluxed with asphalt, a separation of the constituents will take

place, as may be observed by a visual examination of the material under the microscope (Test 2*b*), or sometimes even by the naked eye (Test 2*a*).

German practice favors the use of 15–30 per cent by weight of petroleum asphalt (31–35° C. fusing-point by K. and S. method) and 85–70 per cent by weight of coal-tar pitch.[25]

The effect of successive additions of petroleum asphalt to coal tar generally results in the following changes:

Up to 10 per cent	Slight improvement.
15 to 25 per cent	Marked improvement.
30 to 50 per cent	Segregation and precipitation.
50 to 70 per cent	Increased homogeneity.
70 per cent upwards	Homogeneous and stable.

In preparing the mixture, the asphalt is added to the tar, both being maintained at about 120 to 130° C. The mixture is kept well stirred, while the temperature is gradually increased to 150° C.

In an extreme instance we learn that pulverized oil shale (e.g., Colorado oil-shale) will flux with coal-tar pitch or heavy coal-tar distillates when heated to 300–400° C. until the shale disintegrates and the "kerogen" dissolves.[26] A similar process has been described for fluxing bituminous (i.e., "coking") coal which consists in first pulverizing the coal and then digesting it in a closed retort with coke-oven tar, water-gas tar, or the like.[27] The fluxed mixture may be used for impregnating felt,[28] or for preparing molding compositions.[29]

Hardening the Substance, Raising Its Fusing-point and Increasing Its Stability. Where the bituminous substance is too soft for the purpose intended, it is customary to harden it either by distillation,[30] or by adding one or more of the following materials:

Group I For Hardening Asphaltic Materials	Group II For Hardening Pitches	Group III May Be Used Indiscriminately for Hardening Asphaltic Materials or Pitches
Hard native asphalt	Hard wood-tar pitch	Rosin pitch
Asphaltites	Hard peat- and lignite-tar pitches	Rosin
Hard residual asphalt	Hard water-gas-tar pitch	Hard fatty-acid pitch
Hard sludge asphalt	Hard oil-gas-tar pitch	Blowing with air
Hard wurtzilite asphalt	Hard coal-tar pitch	Combining with sulfur
	Hard bone-tar pitch	Fillers, etc.
		Dispersions of colloidal solids

Of Group I hardeners, hard native asphalts, hard residual asphalts and asphaltites are most frequently used; and similarly of the Group II and Group III hardeners, hard coal-tar pitch and fillers respectively are most generally employed. The fillers may be of vegetable or mineral origin, and will be discussed in greater detail later. It is not customary to harden bituminous mixtures by blowing with air after they are once blended, although this procedure would increase the hardness and particularly the fusing-point. All bituminous substances may be hardened by heating with a small percentage of sulfur after the manner of vulcanization in the rubber industry.[31] This is only used to a limited extent, owing to the difficulty of controlling the degree of hardening, also because of the fact that it tends to reduce the ductility of the product. Other methods for hardening asphalts consist: in heating them with 1 per cent concentrated H_2SO_4;[32] heating natural or petroleum asphalts with 1–10 per cent, or pitches with 5 per cent HNO_3;[33] treating pitches (e.g., naphthol pitch) with mineral acid and formaldehyde;[34] heating asphaltites or asphaltic pyrobitumens under pressure with nascent hydrogen;[35] etc.

Rendering the Mixture Less Susceptible to Temperature Changes. It is difficult to lay down any definite rules in this connection. In general, it may be stated that the suitable addition of the following substances will tend to make mixtures more resistant to temperature changes, viz.:

Asphaltites	Fatty-acid pitch	Rubber
Blown asphalt	Animal or vegetable oils and fats	Chlorinated rubber
Wurtzilite asphalt	Fillers (mineral and vegetable)	

The first three, of course, should only be used in connection with asphaltic mixtures, whereas the next three are applicable either to asphaltic mixtures or pitches. Animal or vegetable oils and fats which have been thickened or "boiled" by heating to a high temperature until they polymerize, are more efficient in this respect than oils or fats in their crude state. Rubber (vulcanized or unvulcanized) or chlorinated rubber[36] may be incorporated in asphalts, coal tars or coal-tar pitches, by: (a) dissolving "crum" rubber in asphalt flux-oil or low-temperature tar-oils, and removing the latter by steam-distillation, and thereupon employing the mixture thus ob-

tained for cutting back harder asphalts or coal-tar pitches; (b) by homogenizing tar with latex, or latex treated with sodium nitrite, and removing the water; (c) by heating asphalt or coal-tar pitch with a mixture containing equal parts by weight of rubber and sulfur. Rubber, when thus incorporated without overheating, will increase the elasticity, hardness and fusing-point of the mixture. If overheated, the rubber will be decomposed and exerts a harmful effect on the product.[37] Rubber may be fluxed with coal-tar pitch in the presence of stearic acid.[38] Other procedures consist in mixing asphalt, coal-tar pitch with cumar (i.e., paracumarone) resin;[39] or phenol-aldehyde resin;[40] incorporating an aqueous mixture of wheat glutin and glycerin and expelling the water under heat;[41] etc.

Increasing the Tensile Strength of the Mixture. For some purposes it is important that a bituminous mixture shall have the maximum tensile strength, to enable it to withstand the stresses and strains to which it may be subjected in usage. This is of special importance in the case of certain forms of bituminous pavements. Two general methods are used for the purpose, viz.:

(1) Incorporating mineral fillers.
(2) Increasing the hardness, by blending with harder bituminous substances.

The tensile strength of a soft bituminous mixture increases to a certain point upon being blended with harder bituminous substances, but these have a tendency to reduce the strength when the mixture reaches the hard and brittle stage.

Making the Mixture More Weatherproof. This is another question which cannot be decided by any hard and fixed rules, as it depends largely upon what materials are present in the mixture, and how badly it may lack weatherproof qualities. There may be cases where the bituminous substance is so deficient in weatherproof properties that it would be impracticable to attempt improving it, on account of the extremely *large proportion* of material which would have to be added to overcome this defect. In general, it may be stated that the addition of the following products tends to overcome the non-weatherproof properties of bituminous mixtures:

GROUP I For Augmenting the Weatherproof Properties of Asphaltic Materials Only	GROUP II For Augmenting the Weatherproof Properties of Pitches as Well as Asphaltic Materials
Asphaltites Certain native asphalts Certain residual oils Blown asphalt Wurtzilite asphalt Certain fatty-acid pitches	Animal or vegetable oils and fats Wool grease Fillers (mineral *only*) Dispersions of colloidal solids

Of the products included in Group I, the asphaltites, wurtzilite asphalt and certain fatty-acid pitches (of the saponifiable type) will most effectively improve the weather-resisting properties of the mixture, due to the fact that these materials of themselves are highly weatherproof. Among the asphaltites, grahamite is most weather-resisting, gilsonite and glance pitch ranging next in efficiency and being about equal in this respect. Wurtzilite asphalt is extremely weatherproof, and the same also applies to the saponifiable varieties of fatty-acid pitch. Only *certain* native asphalts and residual oils are included in this category, for it is impossible to lay down any definite rules to differentiate between the non-weatherproof and weatherproof varieties, since this can only be determined as the result of experience or by an *actual exposure test.* The physical and chemical tests fail to determine definitely whether a native asphalt or residual oil will display the optimum weather-resistance in actual service. Certain tests (e.g., large percentages of volatile matter and non-mineral constituents insoluble in carbon disulfide), may definitely pronounce the material to be non-weatherproof, but if these, by chance, prove negative, the court of final appeal is an *actual* exposure test under service conditions.

Both fluxes enumerated under Group II are equally efficient from the standpoint of weather-resistance, although the first named is superior in its fluxing properties. The use of drying oils combined with metallic oxide driers has also been recommended;[42] also "factice" and cholesterol derivatives;[43] likewise the addition of a small percentage of wood-tar pitch.[44]

Mineral fillers when added in a finely divided state, or in the form of *graded* particles, proportioned to show the minimum percentage of voids, tend to improve the weather-resistance of all bitu-

minous substances. Those fillers which are impermeable to light are most efficient in this respect. The same rules apply in this connection as with mineral pigments in linseed oil paints.[45] It is claimed that the weather-resistance of bituminous substances may be improved by subjecting a warm solution in a solvent to the influence of ultraviolet light, while ozone is being blown through.[46]

Rendering Wax-like, Unctuous to the Feel, or Lessening the Tendency towards Stickiness. For certain purposes, it is desirable to impart the foregoing properties to bituminous mixtures, especially in manufacturing insulating compounds, rubber substitutes, coating compositions for papers, etc. The addition of a small percentage of the following waxes (usually less than 10–15 per cent) will serve to accomplish this result:

Ozokerite **Montan wax** **Pyrogenous waxes**

These will not amalgamate permanently with bituminous materials, but will work their way to the surface in time, forming a thin waxy film which will modify the characteristics of the mixture, imparting certain of the physical properties of waxes. In the case of ozokerite and pyrogenous waxes, only a small percentage should be added, otherwise the separation will be sufficiently great to destroy the integrity of the mixture. Montan wax may be added in large quantities, as it constitutes a better flux and shows but a slight tendency towards separation. The stickiness and tackiness of bituminous mixtures may be decreased by incorporating: a mixture of paraffin wax and phenol;[47] a synthetic resin;[48] resinates, sulfonates, naphthenates, or linoleates of heavy metals (e.g., Mn, Co, Pb, etc.).[49]

Tertiary and Complex Mixtures. In the case of binary mixtures, the characteristics of the blended product may be predicted with a reasonable degree of certainty, but with tertiary or quaternary mixtures this is extremely difficult, and in many cases impossible to do, even by one highly skilled in the art. We must bear in mind that the native asphalts, for example, occur in hundreds of varieties, each differing in certain respects from the others, or as one authority on the subject aptly expresses it: "No two deposits of native asphalt or petroleum on the face of the earth are exactly

alike." Similarly, blown asphalts, residual asphalts, coal-tar pitches, etc., are produced in hundreds of grades, depending upon the nature of the crude materials, the temperature to which they have been subjected, the length of blowing, the duration of the distillation process, and many other factors. These result in the production of a whole series of products from any particular raw material, varying in fusibility, hardness and other physical and chemical characteristics. Since each class of raw material is available in hundreds of varieties, it will be apparent that the number of possible combinations in tertiary mixtures is infinite.

In color matching, a given shade may be produced in a dozen different ways, each starting with totally different colors, and similarly, a given bituminous substance may be exactly duplicated in physical characteristics (i.e., fusing-point, hardness, ductility, tensile strength, volatility, etc.), by numerous mixtures, each containing *different* combinations of *different* materials.

The only way to match a given bituminous substance is by the "cut and try method." This applies with more force when it comes to tertiary and complex mixtures. To duplicate exactly a complex bituminous mixture is one of the most difficult and at the same time one of the most fascinating problems in bitumenology. At the present stage of the science, a chemical analysis of the material to be duplicated will tell nothing. It is only an intimate knowledge of the *physical* properties of the available bituminous raw materials, and an inference of their behavior in combinations, that will assist the expert in synthesizing a mixture having substantially the same properties as the one to be duplicated.

The problem is made still more complicated by the fact that although we may apparently succeed in duplicating the *physical properties* of a given bituminous mixture, yet there is no way of telling other than from an *actual* service test whether or not it will behave the same on *aging* or upon *exposure* to the elements.

Classes of Bituminous Mixtures. Bituminous substances and their mixtures may be roughly divided into three general classes based on their physical properties, characterized by being "soft," "medium" and "hard" at room temperature. The following table will show which of the commercial products belong to the respective classes:

Soft (liquid) Bituminous Products:

Dust-laying oils.
Binders for road surfacings.
Impregnation for wooden paving blocks, railroad ties, etc.
Tars and oils for the flotation process.
Saturating compounds for prepared roofing, flooring, waterproofing, sheathing and insulating papers, electrical insulating tape, etc.
Waterproofing compounds for Portland-cement mortar and concrete.

Medium (semi-liquid to semi-solid) Bituminous Products:

Binders for bituminous surfacings, bituminous macadam and bituminous concrete pavements.
Asphaltic cement for sheet asphalt pavements, asphalt-block pavements and asphalt mastic foot-pavements and floors.
Fillers for wood, brick and stone pavements.
Bituminous expansion joints.
Coatings for prepared roofing, waterproofing, sheathing and insulating papers.
Adhesive compounds for built-up roofing and waterproofing work; plastic compounds for repairing roofs, etc.
Pipe-dips and pipe-sealing compounds.
Electrical insulating compounds.
Rubber substitutes and fillers.
Molding compounds.
Bases of bituminous lacquers and cements.

Hard (solid) Bituminous Products:

Certain forms of electrical insulating compounds.
Molding compounds.
Binders for briquettes.
Certain forms of pipe dips.
Bases of varnishes, enamels, japans and certain bituminous lacquers.

Processes of Compounding Bituminous Substances. The types of apparatus for this purpose fall into two groups, viz.:

(1) Open vessels of semi-cylindrical or rectangular form, as described for dehydrating semi-solid and solid native bituminous substances.

(2) Closed horizontal cylindrical vessels provided with an agitator in the form of a horizontal shaft carrying short stout blades or paddles usually set at an angle. This type is mounted on a masonry foundation over a solid or perforated fire-brick arch, and the heating effected by burning coal or gas underneath it. The vessel is provided with a manhole at the top, through which the bituminous substances are charged, and closed with a cap during the melting process to keep out air and prevent the vapors from igniting.

The first type is used where the bituminous substance is heated below the flash-point of the constituent flashing at the lowest temperature, and the second where it is necessary or desirable to heat the mass above the flash-point. Since the mass can safely be heated in the second type to higher temperatures and agitated at a greater speed without danger of the melted mixture splashing out of the vessel, it follows that with its use the process of amalgamation will take place more rapidly.

The dehydrated bituminous substances are introduced into the melting-tank, preferably in the melted condition, either by gravity or by means of pumps. Where this is not practical, as with high fusing-point products, such as the asphaltites or native asphalts containing a large percentage of mineral matter, they may be added cold in the solid state, but in this case it takes longer to melt up the charge.

The higher the temperature to which the materials are heated, the more rapidly will the combination take place. It is not necessary, or in fact desirable to raise the heat to the fusing-point of the ingredients melting at the highest temperature, as these will be dissolved by the constituents fusing at lower temperatures, due to their inherent *solvent* action combined with mechanical agitation. Thus, a grahamite fusing at 550 to 575° F. (K. and S. method) will readily combine with mixed-base or asphaltic residual oils brought to a temperature of 400° F., particularly if the mixture is kept well agitated. The grahamite should be introduced in the form of lumps about the size of hickory nuts, in preference to a fine powder, as the latter will sinter together if the charge is not agitated, and in addition will make it difficult to tell when the amalgamation is completed.

Great care should be taken not to overheat the bituminous substances, as they are all affected either by a prolonged heating at a moderately high temperature, or upon subjecting to a comparatively high temperature for a short time. There are no general rules regarding the behavior of bituminous substances under the influence of heat. Each will act differently, and resist heat to a greater or lesser degree. It is rarely safe to raise the temperature higher than 450–500° F. in any of the manufacturing processes involving the use of bituminous substances.[50] Overheating will manifest itself by:

(1) Increasing the specific gravity, viscosity, hardness and consistency, fusing-point, flash-point, burning-point, non-mineral matter insoluble in carbon disulfide (free carbon), and carbenes.

(2) Decreasing the ductility, volatile matter, solubility in carbon disulfide and in 88° petroleum naphtha.

On fluxing native asphalts carrying a substantial percentage of mineral matter, it is important to keep the mass well agitated, otherwise the mineral matter will settle out and carbonize against the bottom of the tank, retarding the ingress of heat, and causing the bottom plates to burn out rapidly.

When the mixture is to be heated to a high temperature for other than a comparatively short time, it is inadvisable to effect the agitation by means of air, as this will increase the fusing-point in the same manner as in the production of "blown asphalts." Mechanical stirrers or dry steam jets are preferable under these conditions.

Based upon the uses to which they may be put commercially, bituminous substances may be classified according to their *purity* (i.e., freedom from constituents insoluble in carbon disulfide) as follows:

97-100 per cent purity..	Base of lacquers and varnishes
	Saturants for composition roofings and shingles
	Impregnation for wood, etc.
67- 97 per cent purity..	Coating compositions for roofings
	Adhesive compositions for roofings
	Binders for road surfacings
	Asphaltic cement for sheet-asphalt pavements
18-67 per cent purity...	Molding compositions
	Bituminous expansion joints
	Electrical insulating compounds
	Pipe dips and sealing compounds
12- 18 per cent purity..	Mastic pavements
9- 12 per cent purity..	Sheet-asphalt pavements
5- 11 per cent purity..	Bituminous concrete pavements
Less than 5 per cent....	For enrichment with purer forms of asphalt, etc.

CHAPTER XXIV

BITUMINOUS SUBSTANCES ADMIXED WITH DISCRETE AGGREGATES

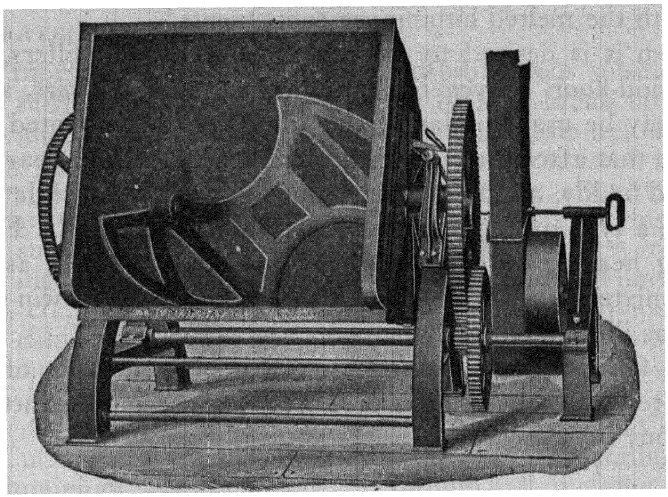
Methods of Incorporating Discrete Aggregates. Since discrete aggregates are incorporated *after* the bituminous mixture has been dehydrated and fluxed to the proper consistency, a steam-heated mixing apparatus of small capacity is best adapted for the purpose, constructed to mix the charge with great rapidity. Two forms of steam-jacketed agitators provided with mechanical mixers are used, viz.:

(1) A rectangular tank with a semi-cylindrical steam-jacketed bottom, commonly provided with two horizontal shafts revolving in opposite directions, each carrying two sets of short strong blades or paddles set at different angles, to work the bituminous mixture from the ends of the vessel towards the centre. The completed mixture is discharged from the bottom through a power-operated slide-valve.

(2) A vertical vessel of cylindrical form provided with a steam-jacketed semi-circular bottom, enclosing a vertical shaft carrying blades, geared to an auxiliary shaft, offset at one end and provided with smaller blades which revolve within the larger ones.[1] The principle of this type is similar to that of a common "egg-beater." The inner shell is cast from a single piece of metal to avoid danger of leakage. This form of apparatus is intended only to mix in such quantities of fillers as will not destroy the fluidity of the mixture or prevent it discharging by gravity through a spout at the bottom. When fillers are used, such as silica, earth-colors, or the like, mixtures may be prepared containing 60 to 65 per cent of the mineral constituents.

It has also been proposed to introduce finely divided fillers in conjunction with a blast of air or steam which is passed through, or directed against the surface of the melted substance;[2] or by introducing the pigment or filler and distilling the mixture to the desired consistency;[3] or by mixing the filler with soft asphalt or flux and

then incorporating same with hard asphalt, rock asphalt or asphaltite;[4] or by introducing a slurry of the filler and kerosene and



Courtesy of J. H. Day & Co.

FIG. 133.—Mixer for Incorporating Large Percentages of Fillers in Asphalt.

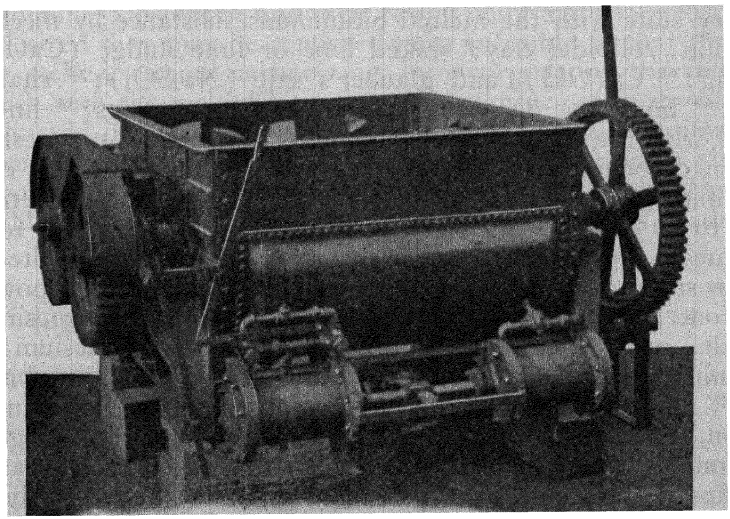


FIG. 134.—Mixer for Incorporating Coarse Aggregates.

then distilling the mixture;[5] or by mixing the filler with an aqueous dispersion of asphalt and then expelling the water by means of

heat;[6] etc. Various devices have been proposed for drying and preheating the mineral fillers and aggregates to facilitate mixing them with the melted bituminous constituents.[7]

When it is desired to incorporate light-weight fillers, such as cork, wood-flour, or vegetable, animal or mineral fibers, a type of mixer may be used similar to the foregoing but mounted on trunnions, so that after the mixing is complete, the entire apparatus may be tipped bodily, and the contents hoed over the rim while the mass is hot in a type of apparatus as illustrated in Fig. 133. For incorporating heavier or coarser form of aggregate, such as used in paving mixtures, a form of batch-mixer, known as a twin-plug mill is used as illustrated in Fig. 134.[8]

The following constitute the more important types of discrete aggregates which have been proposed for use in conjunction with bituminous substances:

(A) COLLOIDAL PARTICLES

1. ADMIXED MECHANICALLY:

(α) **Inorganic.** The following agents may be incorporated in the dry state with the melted bituminous substance by mechanical agitation: colloidal clay;[9] slaked lime or lime sludge ($CaOH_2$);[10] whiting;[11] $Ca(OH)_2$ and glauber's salt (Na_2SO_4);[12] chalk and alum;[13] limestone derived from fresh-water springs;[14] limestone mixed with the melted substance under vacuum;[15] magnesium or barium carbonate;[16] lime soaps (e.g., calcium oleate);[17] calcium cyanamide;[18] lampblack or the like added to asphaltic petroleum prior to its distillation.[19] The following substances in the form of an aqueous suspension, may be incorporated with the melted bituminous substance, and the water then evaporated: clay;[20] powdered limestone or magnesium carbonate;[21] calcium or magnesium hydroxide, calcium sulfate or iron hydroxide;[22] magnesium oxide, aluminium oxide, iron oxide, or silicates of magnesium and aluminium;[23] zinc oxide with acetic acid;[24] etc. In the foregoing connection, it has been found that bituminous mixtures consisting partly of aromatic hydrocarbons (e.g., coal-tar creosote) and partly of paraffins (e.g., semi-asphaltic residual asphalts) act as the best dispersoids, and moreover, the use of all aromatic or all paraffin hydrocarbons will not effect a satisfactory dispersion.

(β) **Organic.** Mixing with soaps (e.g., sodium stearate);[25] or aluminium soap;[26] mixing a hard bituminous substance (e.g., hard coal-tar pitch or peat) with a soft bituminous substance (e.g.,

coal tar) in the presence of a synthetic resin, by means of a colloid mill;[27] etc.

2. LIBERATED "IN SITU":

(α) **Inorganic.** Treating the melted bituminous substance with metallic salts, which under the influence of heat break up into dispersions and either polymerize the bituminous substance, or in some instances combine chemically with same, as manifested by an increase in fusing-point, tensile strength, elasticity, ductility and resistance to fracture. The substances recommended for this purpose include: sulfates or selenates (e.g., ammonium, copper, iron, chromium, manganese, aluminium, sodium, potassium, rubidium, caesium, thallium, etc.);[28] a mixture of copper sulfate and colloidal clay;[29] metallic chlorides (e.g., zinc, aluminium, tin, copper, cobalt, iron, magnesium, titanium, arsenic, antimony and bismuth—the first three giving the best results);[30] metallic oxides and peroxides (e.g., Fe_2O_3, PbO, MnO_2, Ni_2O_3, SnO_2, ZnO, TiO_2, CeO_2, Cr_2O_3, Pb_2O_3, CuO, BaO_2, etc.);[31] borax and borates;[32] metallic acetates (e.g., aluminium, zinc, mercury or sodium);[33] metallic tartrates (e.g., potassium);[34] phosphoric acid;[35] a mixture of boracic and oxalic acids;[36] metallic nitrates; etc.

(β) **Organic.** Decided rubber-like properties may be imparted to bituminous substances, and their fusing-point materially increased, or if desired they may be rendered quite infusible so that they cannot be melted without carbonizing, by incorporating the following mixtures with the melted bituminous substance: casein dissolved in a sulfonated oil, followed by adding formaldehyde;[37] animal glue dissolved in gas-oil;[38] "wrack" dissolved in creosote oil;[39] glycerin-gelatine composition in conjunction with camphor and sulfur;[40] a vegetable phosphatide (i.e., vegetable albumin, such as lecithin);[41] etc.

(B) FILLERS (POWDERS), INCLUDING PIGMENTS

(α) Inorganic

1. *Oxides*:

(a) Silica (SiO_2) in its various forms, including: meta-colloidal silica (e.g., tripoli, trass, volcanic ash, etc.), also hydrated silica (e.g., geyserite);[42] infusorial earth (diatomaceous earth or "Kieselguhr");[43] powdered silica;[44] powdered sandstone;[45] fine sand;[46] waste sand (e.g., from glass works, foundry cores, sandblasting operations, etc.).[47]

(b) Calcium and magnesium oxides (CaO and MgO), in-

cluding: powdered burnt lime;[48] spent lime sludge (e.g., refuse from sugar-refining operations);[49] magnesia oxide;[50] etc.

(c) Aluminium oxide (Al_2O_3), including bauxite and emery (carborundum).[51]

2. *Silicates*:

There are almost infinite varieties of silicates that have been suggested from time to time for use as fillers, including: kaolin or clay;[52] fuller's earth (including spent fuller's earth obtained as a by-product in refining oils);[53] powdered slate;[54] powdered shale, marl or loam;[55] powdered lava;[56] powdered pumice (pumicite) or rottenstone;[57] sillimanite ($2CaO.B_2O_3.2SiO_2$);[58] aluminium silicate or $Ca.Na.Al.SiO_3$;[59] Portland cement;[60] calcareous sandstone;[61] loess;[62] talc or soapstone;[63] asbestos sand, floats and asbestine;[64] microasbestos;[65] mica fines;[66] sea-water mud;[67] soil or earthy matter;[68] etc.

3. *Carbonates*:

In this group there may be mentioned: limestone, chalk or marble dust;[69] crushed sea shells;[70] powdered bones;[71] tufa (tuff);[72] magnesium carbonate;[73] also zinc carbonate.[74]

4. *Sulfates*:

Principally: gypsum (plaster-of-paris or "terra alba")[75] and barium sulfate (blanc fixe).[76]

5. *Phosphates*:

Including: calcium phosphate ("Brewster dust").[77]

6. *Miscellaneous Rock Products*:

This group includes: stone powder ("flour") and dust recovered from crushing operations by air flotation;[78] also electrically precipitated stone-dusts.[79]

7. *Pyrogenous Products*:

In this we find reference to: ground cinders (coal ashes);[80] "fly ash" (recovered from pulverized-coal furnaces);[81] powdered slag;[82] and brickdust.[83]

8. *Black Pigments*:

These include: graphite;[84] graphitic slate;[85] and copper slag (hydrated ferrous silicate).[86]

9. *Colored Pigments*:

Heat-resisting mineral pigments are generally used, including: strong red iron-oxide;[87] micaceous iron-oxide (hematite);[88] yellow ochre;[89] cadmium yellow; green oxide of chromium; ultramarine blue; cobalt blue; titanium oxide; sublimed white lead; lithopone;[90] metallic bronzes (e.g., copper, brass, aluminium, iron, etc.);[91] and mixtures of the foregoing.

(β) Organic

1. *Vegetable Products*:

Including: starch;[92] dust recovered in milling grain;[93] saccharine matter;[94] beet-sugar residues (calcium pectates and calcium carbonate);[95] powdered cork;[96] manure;[97] etc.

2. *Black Pigments*:

Under this sub-division we find: ground peat, lignite or cannel coal;[98] ground oil shales;[99] powdered coal ("culm");[100] pulverized natural rock asphalts;[101] ground coal-tar pitch;[102] lampblack;[103] carbon black and bone black; powdered charcoal;[104] powdered coke (coke breeze);[105] etc.

(C) FIBERS

(α) Inorganic

In this group are included: asbestos;[106] glass wool;[107] slag wool;[108] rock or mineral wool.[109]

(β) Organic

Including: ground or shredded wood (wood wool, wood flour, or cellulose wool), saw-dust, wood shavings, etc.;[110] shredded rag or paper fibers;[111] cotton linters, cotton flock or cottonseed hulls;[112] straw (raw or cooked);[113] flax, ramie, hemp or sisal;[114] wheat bran;[115] rice husks and hulls;[116] corn stalks and sugar-cane fibers;[117] cocoanut fibers;[118] ground tanbark;[119] shoddy and wool waste; hair;[120] ground leather;[121] ground peat;[122] ground roofing scrap and waste;[123] etc. See also "Premolded Expansion Joints," "Fibrated Bituminous Compositions" and "Felt-Making Fibers."

(D) GRANULAR MATTER

(α) Inorganic

Include various groups of "Fillers" given under sections B-α-1 to B-α-7 inclusive, in the granular state; likewise any of the "Inorganic Surfacings—Uncolored Granules" for prepared sheet roofings.

(β) Organic

Include corresponding products referred to under "Premolded Expansion Joints" and "Bituminated Cork Mixtures."

(E) COARSE MINERAL AGGREGATES

1. *Crushed Rock, Stone, Gravel or Slag.* See "Mineral Aggregates" under Paving Materials.
2. *Graded Aggregates.*

(F) COMBINATIONS OF THE FOREGOING

The effect of mineral fillers (Type B-α-3) on the physical characteristics [124] is shown in Table XLVII, based on the mixture of a

TABLE XLVII

Soft residual asphalt (from Mexican oil).....	100%	85%	70%	55%	40%
Precipitated calcium carbonate.....	0%	15%	30%	45%	60%
(Test 9*b*) Penetration at 115° F.....	Too soft	Too soft	Too soft	165	.....
Penetration at 77° F.....	185	135	90	41	.....
Penetration at 32° F.....	60	52	46	39	.....
(Test 9*c*) Consistency at 115° F.....	0.0	0.0	0 0	3.1	.....
Consistency at 77° F.....	3.1	4.6	6.5	13.9	.....
Consistency at 32° F.....	18.4	22.2	24 1	30.4	.....
(Test 9*d*) Susceptibility index	19.8	22.6	19 5	14.5	.....
(Test 10*b*) Ductility at 115° F.....	14.0	13.0	10.0	2.5	.....
Ductility at 77° F.....	40 0	31 0	13.0	0 6	.....
Ductility at 32° F.....	2.7	2.6	0.8	0.5	.....
(Test 11) Tensile strength at 115° F	0.0	0.0	.0.0	0.3	.....
Tensile strength at 77° F.....	0.4	1.1	1.7	2 4	.....
Tensile strength at 32° F.....	5.5	7.5	9 5	13.0	.....
(Test 15*a*) Fusing-point (K. and S. method)	93° F.	98° F.	106° F.	163° F.	.....
(Test 15*b*) Fusing-point (R. and B. method)	110° F.	115° F.	124 °F.	188° F.	.....
Medium residual asphalt (from Mexican oil)	100%	85%	70%	55%	40%
Precipitated calcium carbonate....	0%	15%	30%	45%	60%
(Test 9*b*) Penetration at 115° F.....	240	170	80	27	20
Penetration at 77° F.....	60	47	32	18	9
Penetration at 32° F.....	19	16	13	9	5
(Test 9*c*) Consistency at 115° F.....	2.1	2.7	5.75	13.6	17.1
Consistency at 77° F.....	10.0	12.4	17.65	26.2	36.6
Consistency at 32° F.....	47.3	51.9	54.3	66.9	80.4
(Test 9*d*) Susceptibility index.....	38.2	39.7	35.9	28.0	26.2
(Test 10*b*) Ductility at 115° F.....	27.0	22.0	11.0	0.8	*
Ductility at 77° F.....	38.0	13.0	1.1	0.25	*
Ductility at 32° F.....	0.0	0.2	0.1	0.0	*
(Test 11) Tensile strength at 115° F.....	0.0	0.2	0.8	1.8	*
Tensile strength at 77° F.	1.6	2.2	4.3	6.7	*
Tensile strength at 32° F.....	9.6	14.0	20.5	27.2	*
(Test 15*a*) Fusing-point (K. and S. method).	118° F.	124° F.	135° F.	190° F.	242° F.
(Test 15*b*) Fusing-point (R. and B. method).	136° F.	145° F.	158° F.	216° F.	269° F.

* Does not flow when heated.

straight run residual Mexican asphalt with 0, 15, 30, 45 and 60 per cent of precipitated calcium carbonate.

It will be noted that the fusing-point, hardness, and tensile strength increase, whereas the susceptibility index and ductility decrease in proportion to the quantity of filler added. When the percentage of filler is sufficient to destroy the fluidity of the mixture, as in the case of the surface course of sheet asphalt pavements (where the filler exceeds 80 per cent by weight), the physical properties of the mixture depend largely upon the pressure to which the mass has been subjected. The greater the compression, the greater will be the hardness, tensile strength and density.

Mixing increasing percentages of fillers with coal-tar pitch has the following effects:

(1) The percentage loss on evaporation (figured on the weight of coal-tar pitch in the mixture) is progressively decreased.

(2) The fusing-point of the mixture is increased in direct proportion to the degree of fineness of the filler, irrespective of its chemical composition.

(3) The difference between the fusing-point of the mixture, before and after the evaporation test, is progressively decreased.

(4) The Ubbelohde dropping-point (Test 15*h*) of the mixture is progressively increased.

(5) The difference between the dropping-point of the mixture, before and after the evaporation test, is progressively decreased.

(6) The "breaking-point" (Test 13) of the mixture is progressively decreased.

(7) Different fillers influence the characteristics of the mixture to a greater or lesser extent, depending upon their physical characteristics, degree of subdivision, etc.

(8) Mixtures of two different fillers often give results superior to the use of either filler alone.

Tests for load characteristics, compressibility and mechanical strength, show the fillers named in the order of their superiority for use in tar-concrete pavements: limestone, copper slag, sandstone, Portland cement, volcanic slag, slate, etc.[125]

The effect of inorganic colloidal particles liberated "in situ" (Type A-2-α) is shown in the following figures, obtained upon heating 1 kg. of residual oil at 400° F. for one hour with the metallic salt named:

TABLE XLVIII

	Fusing-point (K. and S.) (Test 15*a*)	Penetration at 77° F. (Test 9*b*)	Hardness at 77° F. (Test 9*c*)
Semi-asphaltic Residual Oil:			
Untreated	102° F.	120	4.93
With 2 per cent ferrous sulfate	105	108	5.57
With 2 per cent aluminium chloride	114	81	7.37
With 2 per cent copper sulfate	118	74	8.09
With 2 per cent copper carbonate	118	70	8.48
With 2 per cent zinc chloride	130	44	13.2
With 3 per cent zinc chloride	142	32	17.4
With 4 per cent zinc chloride	162	20	24.1
With 5 per cent zinc chloride	168	19	24.7
Asphaltic Residual Oil:			
Untreated	105	115	5.0
With 2 per cent copper sulfate	128	68	8.9
With 3 per cent copper sulfate	131	60	10 0
With 4 per cent copper sulfate	141	51	11.7
With 5 per cent copper sulfate	155	43	13 7
With 2 per cent zinc chloride	140	50	11.9
With 3 per cent zinc chloride	176	39	15 0
With 4 per cent zinc chloride	205	31	18 0
With 5 per cent zinc chloride	217	27	19 5

After treatment, the first series showed a bright lustre and the second series a dull lustre.

CHAPTER XXV

BITUMINOUS SUBSTANCES DISPERSED IN WATER

Types of Bituminous Dispersions

These include bituminous-aqueous emulsions and suspensions. A bituminous-aqueous *emulsion* consists of a dispersion of a *liquid* or *semi-liquid* bituminous substance throughout water, and a bituminous-aqueous *suspension* consists of a dispersion of a *semi-solid* or *solid* bituminous substance throughout water. A bituminous emulsion constitutes two liquid phases—a bituminous phase and an aqueous phase— a "phase" being regarded as a physically distinct, mechanically separable part of the system. A bituminous suspension constitutes a non-fluid or solid bituminous phase and a liquid phase. Systems in which either a solid or a liquid is dispersed in a liquid medium, are termed "sols," and if the liquid medium is aqueous, the colloidal system is termed a "hydrosol," whereas if the liquid medium is an oil, the system is termed an "oleosol." The agglomeration, coalescing and deposition of the disperse phase is termed "coagulation" or "peptization." The coagulation of sols may be in the form of more or less definite particles, or of a jelly, or of a flaky mass, usually termed "flocculent." The precipitate formed is called a "gel." The dispersed particles are termed "micelles" if they have absorbed another constituent on their surface, with which they combine as the disperse phase. A substance which renders dispersions stable, once they are formed, as well as to facilitate their formation, is termed a "dispersing agent," or a "deflocculating agent," or a "peptizing agent." This is known as a "protective colloid" if a solid, and an "emulsifying agent," or "suspending agent," or "stabilizing agent," if a liquid. The bituminous-aqueous dispersion is said to have the bituminous constituent in the "external" or "continuous phase" and the water in the "internal" or "discrete phase," when the dispersion will mix readily with more of the

bituminous constituents but not so readily with water. The bituminous constituent is said to be in the "internal" or "discrete phase," and the water in the "external" or "continuous phase," when the converse is true. However, it is possible to produce bituminous-aqueous dispersions which will mix with either water or more of the bituminous constituents with equal facility.

Certain colloids, when desiccated, will become re-dispersed upon the addition of the dispersion medium, without any special manipulation being required. Such colloids are termed "reversible," and when the continuous phase is aqueous, are said to be "hydrophile" or "lyophile" (i.e., water-loving) because of their ready re-dispersion in water. On the other hand, other colloids, on desiccation, do not manifest this property, and are termed irreversible and are said to be "hydrophobe" or "lyophobe" (i.e., water-fearing) when the continuous phase is aqueous.

When a suspension and an emulsion are mixed, the non-fluid or solid particles in the former coalesce with the liquid globules of the latter, and viscous liquid or semi-solid globules are formed which are prevented from coalescing by the protective colloid or emulsifying agent, and thereby form a stable dispersion. Thus, although the dispersing agent prevents the viscous globules from coalescing with each other, it does not prevent the solid or non-fluid particles from coalescing with the fluid globules.

Investigations on surface- and interfacial-tension enable us to understand how dispersing agents effect a stabilization of aqueous-bituminous dispersions. For example, at 20° C., the interfacial tension between water and benzol is 35.0 dynes per cm. If, however, the water contains 0.4 per cent NaOH and the benzol 2.8 per cent of oleic acid, the interfacial tension is reduced to 0.04 dyne per cm., or about 0.001 of the initial value. As an empirical rule, it has been shown that dispersions are stabilized by substances forming colloidal solutions or suspensions in water. The majority of dispersions contain globules of varied dimensions, with the smaller globules squeezed into the spaces between the larger ones. The dispersing agent serves to inhibit the coalescence of the internal phase and does not necessarily determine the degree of dispersion attained. After evaporation of the water, the dispersing agent remains in the bitumen as a "filiform," or honeycomb structure, which

serves to modify the physical properties of the film (i.e., fusing-point, gloss, weather-resistance, etc.).[1]

Forms of Apparatus Used. Two types of apparatus are employed to produce bituminous-aqueous dispersions:

(1) Agitators or "homogenizers."
(2) High-speed disintegrators, termed "colloid mills."

Agitators are adapted to treating liquid or semi-liquid bituminous substances which may be readily broken up in the cold into minute globules. Semi-solid or solid bituminous substances may also be dispersed by means of agitators, if treated in the heated state, and provided they melt below the boiling-point of water. Similarly, solid bituminous substances may be dispersed in this type of apparatus, provided they are first comminuted by grinding, atomizing,[2] or the like, and the powder agitated in the cold with cold water containing the dispersing agent. A typical form of apparatus consists of a jacketed vessel in which is suspended a metal cylinder open at both ends, enclosing a vertical shaft carrying propellers adjustable to varying heights on the shaft. By reversing the pitch of the lower propeller, a powerful pull is created from the bottom of the mixing vessel, while the upper propeller creates a downward flow. The two opposing streams are thus forced against each other and then thrust against the sides of the vessel with great force. In this case the dispersion is the result of violent turbulence. A variation consists in using a revolving cylinder fitted with hollow trunnions carrying a horizontal, revolving shaft with a number of beaters arranged to run in the opposite direction to that of the cylinder and its contents. In all cases, it is desirable to run the disperse phase into the external phase, so as to assist mechanically the promotion of the dispersion. The dispersing agent is preferably carried in solution or suspension in the external phase. The water containing the dispersing agent is first introduced through a pipe, and while the propellers are in motion, the bituminous constituents are gradually run in through another pipe, either in the cold (if sufficiently fluid), or else in a molten condition.[3] The finished emulsion is drawn off at the completion of the operation and cooled rapidly to room temperature.[4] Thus, in the case of an asphalt having a fusing-point of 150° F. (R. and B.) and a penetra-

tion of 28 at 77° F., and with the use of clay as dispersing agent, the following procedure is recommended: 30 parts of clay and 70 parts of water are introduced at 120° F.; then the asphalt at 220° F. is gradually added under continuous stirring. The completed emulsion will consist of 6 per cent clay, 14 per cent water and 80 per cent asphalt.

Other forms of agitators may consist of masticators, agitators, ball-mills, pug-mills, chasers or chilean mills, etc.,[5] in which mechanical agitation is coupled with a certain amount of grinding or trituration. These forms of apparatus are no longer used, as they are comparatively inefficient. It may be stated in general, however, that the efficiency of the agitator will depend upon the amount of power or energy expended on the mixing operation.

Colloid mills have now supplanted the mechanical type of agitators, since they not only form a better dispersion, but also consume a smaller amount of dispersing agent, or even it is claimed, without necessitating the presence of any dispersing agent whatever.[6] In this form of apparatus, the dispersion is obtained by providing additional mechanical energy in substitution of part, or all of the dispersing agent. They are adapted to treating liquid or comminuted solid substances in the cold, as well as all the types of bituminous substances in the heated state, provided they melt at, or near, the boiling-point of water. The colloid mill consumes considerably more power than agitators, which accounts for its greater efficiency in breaking up the bituminous substance into small globules or particles.

Colloid mills effect a dispersion resulting in particles rarely less than 1μ in diameter, which are actually larger than what is generally accepted as collodial dimensions, involving a particle diameter not exceeding 200 mμ. Hence it is more accurate to refer to this type of apparatus as a "dispersion mill" rather than a colloid mill. Furthermore, a sharp distinction exists between a "homogenizer" which operates under a pressure of 2000 to 3000 lb. per sq. in. and the colloid or dispersion mill, since the homogenizer is not adapted for dispersions of solids or heavy liquids. Although some pressure is utilized in the colloid mill, the main effect depends upon the hydraulic shearing forces exerted upon the particles in the presence of a fluid by the rotating surfaces. In the case of asphalts,

the size of the dispersed particles is rarely less than 1μ; usually about 3-6μ; the upper limit being about 15μ in diameter. The particles in the neighborhood of 1μ show Brownian movement, which is absent in the larger particles. In the presence of a stabilizing agent, the dispersions carrying the larger particles will remain stable—otherwise they are apt to "break" if the particles average greater than 5μ (i.e. in the absence of a suitable stabilizing agent).[7]

There are two general types of colloid mills,[8] viz.: beater-type mills, in which the particles are subjected not only to hydraulic shearing stresses, but also to impact stresses produced by revolving blades operating between fixed blades. This operates on the batch system and includes the original Plauson machine and its subsequent modifications,[9] including the Hurrell mill.[10]

The second consists of the smooth-surfaced type, in which two smooth surfaces in fairly close contact are rotated at a peripheral velocity of 2 miles per minute upward, and a low-pressure pump feeds the mixture between the disintegrating elements which consist either of a truncated cone rotating within a fixed cone, as illustrated in Fig. 135,[11] or two flat discs rotating in opposite directions, positioned either vertically, or horizontally as illustrated in Fig. 136.[12] In the latter type of machine, the material is introduced through the shaft attached to one of the discs, being thrown off the periphery of the discs into a chamber where it is removed by gravity. In some cases the discs are roughened, so as to augment the hydraulic shearing action. The moving elements have a clearance of 0.002-in. upwards and are operated at speeds of 3500 r.p.m. upwards. For each type of mill there exists an optimum speed, or degree of agitation or mixing, and an optimum period of running-time, whereby the most stable dispersion may be obtained for a given system. In general, there is a speed above which the stability of the dispersion decreases. The practical significance of this is obvious, since an excessive expenditure of power is unnecessary.

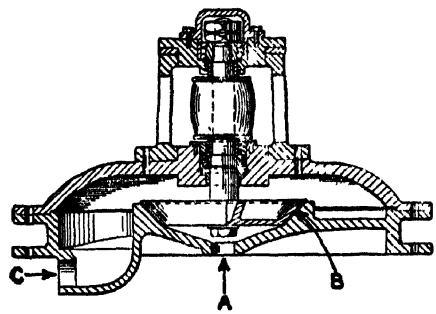


FIG. 135.—Smooth-surface Type of Colloid Mill with Truncated-cone Working Surfaces. *A*—Material inlet; *B*—Working surfaces; *C*—Material outlet.

Solids should always be subjected to a preliminary grinding to not less than 80 mesh, and generally finer, depending upon the clearance at which the mill operates. A form of mill has been proposed to accomplish this, consisting of a pair of rotating discs with projecting vanes or pins.[13]

Among the factors which influence the dispersion are temperature, hydrogen-ion concentration and the protective colloid or dispersing agent used.

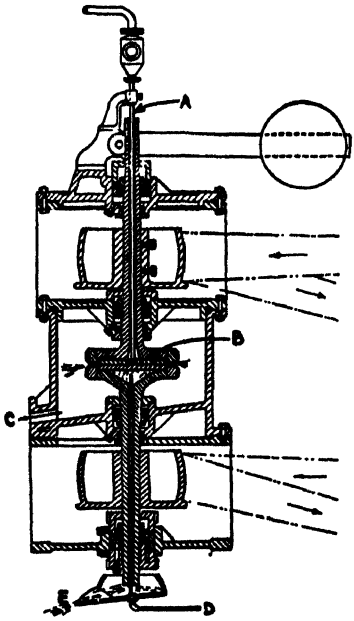


FIG. 136.—Smooth-surface Type of Colloid Mill with Opposed-disc Working Surfaces. *A*—Material inlet; *B*—Working surfaces; *C*—Material outlet; *D*—Cooling-water inlet; *E*—Cooling-water outlet.

The effect of rise in temperature is in general to make the dispersion easier. For non-miscible liquids, rise in temperature is accompanied by a decrease in the interfacial tension, a condition favorable to dispersion. A colloid mill will effect the dispersion with a smaller amount of dispersing agent than if merely agitated mechanically, as may be illustrated by the following figures: 100 parts of water are heated to 90° C. and 3.5 parts caustic soda and 25 parts oleic acid are added. The resultant mixture, containing 20 per cent by weight of sodium oleate is diluted with 9 times its volume of water heated to 90° C. and then run through a colloid mill with an equal amount of asphalt heated to not exceeding 100° C. The resulting emulsion contains equal parts of asphalt and water, with 1 per cent by weight of soap, or in other words 50 times as much asphalt as soap.[14] On the other hand, by using an agitator of the average efficiency, the best that may be accomplished with the same ingredients is to form an emulsion with about 25 times as much asphalt as soap, even though the former is introduced in installments.[15]

Character of the Bituminous Substance. They may either be soft or hard. Soft substances may be dispersed at room temperatures, or with the application of a moderate amount of heat. Hard

substances may either be pulverized and dispersed at room temperature, or else melted and dispersed in the fluid state. Practically every form of bituminous substance has been proposed for dispersions, including: natural and artificial asphalts; coal tar and coal-tar pitch; various mixtures, such as asphalt fluxed with low-temperature tar.[16]

Character of the Dispersing Agent. The following constitute the principal types of dispersing agents which have been proposed from time to time for use with bituminous substances.[17]

(A) INORGANIC SUBSTANCES

(1) *Hydroxides.* Including: $FeOH_3$, $AlOH_3$, $CaOH_2$, $BaOH_2$, etc.[18]

(2) *Oxides.* Including: Fe_2O_3, Al_2O_3, CaO, MgO, BaO_2, ZnO, CuO, Pb_3O_4, etc.;[19] various forms of silica, such as silica gel (i.e., hydrated silicic acid);[20] infusorial earth;[21] sand.[22]

(3) *Silicates.* Including: soluble silicates (e.g. Na_2SiO_3);[23] insoluble silicates (e.g. Fe, Al, Ca or Mg—such as talc, soapstone, hydrous magnesium silicate, etc.);[24] colloidal clays[25] inclusive of bentonite;[26] spent fuller's earth;[27] Portland cement;[28] soil or earthy matter;[29] etc.

(4) *Sulfates.* Including gypsum (plaster-of-Paris).[30]

(5) *Phosphates.* Such as tri-sodium phosphate or sodium-hydrogen phosphate.[31]

(6) *Sulfides or Polysulfides.* Such as Na_2S or K_2S (with or without Na_2CO_3);[32] arsenious sulphide;[33] etc.

(7) *Alkalies.* These form "soaps" with bituminous substances containing saponifiable constituents (e.g., wood tar, rosin pitch, lignite tar, montan wax, fatty-acid pitch, certain coal tars, certain asphalts, etc.), and include: NH_4OH, KOH, NaOH, K_2CO_3, Na_2CO_3, borax, etc.;[34] alkalies plus an asphalt solvent (e.g., benzol).[35]

(8) *Miscellaneous.* Including calcium arsenate, basic cadmium sulfate, etc.

(B) ORGANIC SUBSTANCES

(1) *Fats and Oils.* Such as cholesterol alone;[36] sulfonated cholesterol;[37] cholesterol with H_3BO_3 or H_3PO_4;[38] stearic acid with diethylene or triethylene glycol.[39]

(2) *Resins.* As for example synthetic resins (e.g. phenol-aldehyde, or cumarone) with or without alkali.[40]

(3) *Soaps.* Including combinations of NH_3, K, Na, Mg, and Ca, with fatty acids or resin acids (e.g., fats, fatty-acids, saponifiable oils, cholesterol, fatty-acid pitch, wood tar, wood-tar pitch, rosin, rosin pitch, montan wax, lignite tar, etc.);[41] ammonium linoleate or trihydroxy-ethylamine stearate, sulfates, etc.;[42] triethanolamine oleate;[43] glycerinmonostearic-acid ester and amino-ethyleneamido-chlorhydrate;[44] soap and cyclohexanol;[45] crude lignite wax and alkali;[46] etc.

(4) *Sulfonated Vegetable Oils.* Including turkey-red oil;[47] metal sulfonates (with glycerin, etc.).[48]

(5) *Sulfite Liquor* (derived from alkali-cellulose manufacture). Includes the use of sulfite liquor alone;[49] with slaked lime;[50] with Fe, Al or Cr sulfate;[51] also sulfite-cellulose pitch.[52]

(6) *Mineral-Oil Derivatives.* These include polymerides of olefin (i.e. alkaline oxides);[53] naphthenic acids and their salts (i.e. sulfo-derivatives, acid sludges, oxidation products, etc.);[54] aromatic sulfo-derivatives (e.g. "kontakt");[55] oxidized paraffin wax with or without NaOH;[56] oxidized mineral oil products (e.g. blown with Na_2CO_3 and NH_4-stearate);[57] chlorinated mineral oil derivatives;[58] residues from cracking processes (e.g. "Dubb's Sludge");[59] etc.

(7) *Carbonaceous Matter.* Such as powdered hard bituminous substances;[60] powdered lignite, brown-coal or oil shale;[61] powdered bituminous coal;[62] carbon black;[63] etc.

(8) *Alkaline Bases.* Including pyridines, amines (e.g. triethanolamine), cresylates, piccolin, quinolin, etc.;[64] resorcinol, pyrogallol, α or β-naphthol, carbazol, etc.[65]

(9) *Proteins or Proteids.* Include alkaline caseinates, albumin, lecithin;[66] vegetable proteins (e.g. soya-bean flour);[67] mucilaginous extract from linseed or flaxseed hulls.[68]

(10) *Albumenoids.* Such as glues and gelatines.[69]

(11) *Pectins.* Include the various gels.[70]

(12) *Gums and Algae.* Such as gum-Arabic (acacia), tragacanth, various seed-gums, agar-agar, Irish-moss (carrageen moss) etc.;[71] algin;[72] alkaline alginates (e.g. seaweed and alkali);[73] etc.

(13) *Polysaccharides and Hemicelluloses.* Such as starch, dextrine, glucose and soluble carbohydrates, with or without alkali;[74] molasses residues with $CaOH_2$;[75] etc.

(14) *Tannins.* Include tannic acid, alkaline tannates, heavy metal tannates, various bark extracts, saponin (soap bark), lignins, humic acid and alkaline humates (from turf, peat, brown-coal, decayed vegetable matter, etc.).[76]

(15) *Miscellaneous.* Include glycerin residues obtained on distilling glycerin;[77] viscose;[78] bile salts (e.g. oxgall);[79] β-tetrahydronaphthalene;[80] etc.

(C) VARIOUS COMBINATIONS

(1) *Clay in Combination With*: Inorganic hydroxides;[81] alkaline silicates or metal fluosilicates;[82] Portland cement;[83] alum or sodium-hydrogen phosphate;[84] phosphoric, chromic, oxalic or benzoic acid;[85] alkalies;[86] soaps of various kinds;[87] sulfite liquor;[88] alkaline caseinates;[89] glue or gelatine;[90] starch paste;[91] tannic acid or alkaline tannates;[92] tri-cresyl phosphate;[93] etc.

(2) *Sodium Silicate in Combination With*: Slaked lime ($CaOH_2$);[94] or rosin.[95]

(3) *Soaps in Combination With*: Alkaline silicates;[96] sodium-hydrogen phosphate;[97] borax;[98] sulfonated vegetable oil;[99] phenol-sulfonic acid;[100] alkaline caseinates;[101] glue or gelatine;[102] gums (e.g. algae jelly or carrageen moss);[103] starch paste (or farinaceous protein);[104] tannic acid or alkaline tannates;[105] etc.

(4) *Sulphonated Vegetable Oils in Combination With*: Alkaline caseinates;[106] glue.[107]

(5) *Alkaline Caseinates in Combination With*: Agar-agar;[108] alkaline tannates;[109] the latter it is claimed will form exceedingly fine-grained dispersions which will not "break" readily even when ferrous sulfate is added.

(6) *Glue or Gelatine in Combination With*: Aluminium salts (e.g. $AlOH_3$ or aluminium acetate);[110] molasses;[111] etc.

(7) *Tannic Acid or Alkaline Tannates in Combination With*: Starch paste;[112] tri-sodium phosphate;[113] etc.

Slow-setting, "stabile" dispersions are prepared with the use of inorganic hydroxides, clay, and other water-insoluble dispersing agents. Such dispersions will not break upon adding electrolytes, inorganic fillers (e.g. sand, silicates, asbestos, Portland cement, pigments, etc.), or organic fillers (e.g. wood flour, cork powder, etc.). Quick-setting, "labile" dispersions are prepared with the use of soaps and other water-soluble dispersing agents. Such dispersions will break rapidly upon adding inorganic or organic fillers. The speed of setting may be regulated by mixing together water-insoluble and water-soluble dispersing agents in suitable proportions; or by mixing together a rapid-breaking dispersion with a slow-breaking variety.[114] The use of dispersing agents in verious combinations (Group "C") not only gives better results than the use of one agent alone, but also effects a saving in the quantity required to accomplish a given result. Water-soluble dispersing agents are known as "stabilizing agents."[115]

The addition of the following materials is claimed to retard the breaking of dispersions: an acid in the presence of Na or K fluoride, oxalate, tartrate or fluosilicate;[116] an acid dye-stuff with potassium ferrocyanide;[117] basic dyes;[118] sodium zincate or sodium aluminate;[119] dissolved gases (e.g. acetylene);[120] etc.

The addition of the following substances is claimed to induce bituminous dispersions to break more rapidly: electrolytes, such as H_2SO_4, HCl, H_3PO_4, H_3CrO_4; likewise $CaCl_2$, NaCl, Na_2SO_4, etc.[121]

The mobility of bituminous dispersions may be increased by adding a small percentage of citric or mineral acids.[122] On the other hand, the viscosity may be increased by adding: a carbohydrate, a phenol, or a colloid capable of swelling and forming a gel.[123]

A previously-formed dispersion may act as dispersing agent for additional bituminous ingredients, with or without the addition of dilute alkali as the external phase.[124]

Asphalt-solvents may be incorporated with bituminous dispersions, especially in preparing emulsion paints (see "Bituminous Emulsion Paints"). In certain cases tar dispersions are prepared with 10-20 per cent by weight of hydrocarbon solvents in addition to 40-50 per cent by weight of water.

To prevent bituminous dispersions from congealing at low temperatures, the addition of the following agents has been suggested for the purpose of lowering the freezing-point of the aqueous phase: methyl alcohol;[125] aliphatic monohydric alcohols;[126] polyhydric alcohols;[127] acetone;[128] glycerine;[129] glycol, aniline, furfurol, etc.;[130] alkaline thiocyanates;[131] etc.

Uses of Bituminous Dispersions. Dispersions of bituminous substances with water are utilized commercially for the following purposes:

(1) Paving, including dust-laying, road-surfacing, cold patching and in certain cases as the binding medium for bituminous macadam pavements.

(2) Impregnating paper, felt and their respective pulps, and in certain cases for coating the same.

(3) As a substitute for coal-tar pitch or asphalt as the cementing medium for constructing built-up roofs.

(4) As a waterproofing agent for rendering Portland-cement mortars and concrete impervious to water, also as a surface application to concrete to prevent the premature evaporation of water during the curing process, in which case 1 gal. is applied per 6 sq. yds. followed by an application of sand or grit.[132]

(5) For replacing the solvent in manufacturing bituminous lacquers, japans and plastic compositions.

(6) For surfacing cement floors as an equivalent of asphalt-mastic flooring compositions in conjunction with a mineral aggregate.[133]

(7) For heat insulation purposes, as a binder for cork in constructing walls and ceilings.

(8) For coating underground pipes and preventing the corrosion of metals. In this case the addition of chromium salts will impart anti-corrosive properties to the dispersion.

(9) For manufacturing electrodes, etc.

(10) Manufacture of rubber substitutes, in which case the bituminous emulsion may be readily mixed with rubber latex,[134] gutta-percha pitch, etc.

(11) For use in tree surgery.[135]

(12) As a sound-deadener when applied on the inside of metal automobile bodies, for which purpose it is either sprayed on as such, or admixed with fillers or fibrous matter (with or without solvents).[136]

(13) As a lubricant for hot bearings, etc.[137]

(14) As an insecticide or fungicide, in which case it is mixed with As_2O_3, or the like.[138]

(15) For filling cavities in structures or in the ground.[139]

(16) As a binder for briquettes.[140]

CHAPTER XXVI

BITUMINOUS SUBSTANCES DISSOLVED IN SOLVENTS

Classes of Solvents. The principal commercial solvents of interest in connection with bituminous substances fall into the following groups:

1. Hydrocarbons:
   - (*a*) Derived from petroleum (aliphatic).
   - (*b*) Derived from wood-distillation ("naval stores' solvents").
   - (*c*) Derived from coal-distillation (aromatic).
2. Chemical products:
   - (*a*) Sulfur derivatives.
   - (*b*) Chlorine derivatives.
   - (*c*) Nitrogen derivatives.
   - (*d*) Aceto derivatives.
   - (*e*) Alcohols.
   - (*f*) Ethers.
   - (*g*) Esters.
   - (*h*) Ketones.

Table XLIX gives the characteristics of the principal solvents in Classes 1 (*a*), 1 (*b*) and 2; and Table L those in Class 1 (*c*).

Function of Solvents. Among the objectives to be attained through the use of solvents in conjunction with bituminous substances, the following may be cited:

1. A *complete* solution of the bituminous substance,

(*a*) whereby the bituminous substance is purified by being brought into complete solution in the solvent and extracted, or otherwise separated from any associated non-bituminous constituents (e.g. mineral aggregate, carbonaceous matter, or other insoluble organic constituents). In this case, the solvent is subsequently recovered by distillation and the bituminous substance separated in its purified state.

(*b*) whereby the bituminous substance is liquefied upon being dissolved in the solvent and thereby transformed into a lacquer, or varnish, or plastic composition, which is adapted to being applied

to objects in the cold by means of a brush, a spray-gun, a trowel, or the like. In this case, the solvent simply acts as a vehicle and evaporates spontaneously after application, either in the air, or upon being baked in an oven.

2. A *partial* solution and/or precipitation of the bituminous constituents through the selective action of a particular solvent, or mixture of solvents, whereby certain of the bituminous constituents are brought into solution and the remainder are precipitated. In this manner the physical properties of the bituminous substance may be suitably modified through the separation of the less desirable fractions from those having the optimum characteristics. As in the first case, the solvent is recovered by distillation and used over again.

Behavior of Solvents. It is a well-known fact that different solvents exert varying degrees of solvent power on bituminous substances, which may be attributed to variable physical effects, including:

(1) Absolute differences in true solvent power.

(2) The temperature at which the solvent is permitted to act. It may be stated generally in this connection that all solvents exert a greater solvent effect at high temperatures than at low temperatures.

(3) Ability of certain solvents to retain material in colloidal suspension, which would otherwise manifest itself as insoluble—termed a "peptizing" reaction. Conversely, the effect of certain solvents to coagulate material which was originally maintained in colloidal suspension—termed a "flocculating" reaction.

(4) Adsorption of the solvent, resulting in the formation of insoluble compounds, which separate out or "flocculate."

(5) Adsorption of the bituminous constituents by mineral constituents present in the mixture.

It has been ascertained that solvents having a low surface-tension act as flocculating agents, whereas those having a high surface-tension behave as peptizing agents. A certain relationship is assumed to exist between the surface-tension of the bituminous substance and that of the solvent, the addition of which serves to alter the interfacial tension of the medium-micelle, and may result in the flocculation of the micelle. This may occur rapidly and completely, or may continue slowly over an extended period of time.[2]

The following solvents have been rated according to their sur-

TABLE XLIX

Characteristics of Various Commercial Solvents [1]

Name	Formula	Mol. Wt.	Distilling Range*		Melting Point† ° C.	Flash Point‡ ° C.	Vapor Pressure at 30° C. *Mm. Hg.*	Sp. Gr. at 20° C.††	Wt./Gal. at 20° C. Lb.	Refractive Index at 20° C.†	Expansion Coefficient, 10–30° C.	Purity Commercial Product %
			Initial b. p. ° C.	End point ° C.								
HYDROCARBONS												
(*a*) *Derived from Petroleum:*												
Petroleum naphtha		....	100	130	.....	>30	§	0 735	6 12	.....	0 00115	.....
Mineral spirits		....	145	210	... .	>40	§	0 750	6 25	.....	0.00098	.....
High-test naphtha		....	168	230	.....	>50	§	0.765	6.37	.....	0.00098	.....
Kerosene		....	±90% to 300°		.....	100–120	.....	0.775–0 850	6 46–7 08	.....	.....	.....
Gas oil		...	±90% to 400°		. . .	150–225	....	0 825–0 900	6 88–7 50	. .....	.....	.....
(*b*) *Derived from Wood:*												
Gum spirits of turpentine		....	154	201	.....	110	§	0.868	7.22	.....	0.00090	.....
Steam-distilled turpentine		. . .	155	173	.....	115	§	0 857	7.13	.....	0.00089	.....
Dipentene	$C{\cdot}CH_3(C_4H_7)C{:}(CH_3)_2$	136.13	172	200	.. ...	145	2	0 853	7 10	1.473	0 00086	.....
Hercosol		.. .	176	262	.....	150	§	0 912	7 59	.....	0.00083	.....
Pine oil		....	99	226	.... .	185	§	0.925–0 935	7 7	.....	0 00066	.....
Heavy pine oil		....	211	219	.. ...	200	§	0 937–0 942	7 8	....	0 00085	.....
Fenchone	$(CH_3)_2C_6H_7C{:}O$	153 17	190	210	6	160	2	0.948	7 89	1.4647 (14.5° C.)	0 00088	.....
Furane derivatives:												
Furfural	$(C_4H_3O){\cdot}CHO$	96 03	158	162	−38.7	145	5	1 159	9 64	1.5261	0 00087	98
Furfuryl acetate	$CH_3COOCH_2(C_4H_3O)$	140 06	180	186	.. ...	185	1	1 119	8.42	1 4627	0 00094	99
Furfuryl alcohol	$C_4H_3OCH_2OH$	98 05	152	220	...	175	2	1.137	9 46	1 4852 (22.7° C.)	0 00077	98
Tetrahydrofurfuryl alcohol	$C_4H_7OCH_2OH$	102.08	119	204	.. . .	180	<1	1 051	8 74	....	0 00074	99
(*c*) *Derived from Coal:* See Table L.												
Cyclohexane	C_6H_{12}	84 09	80	83	6.5	<6.5**	121	0 779	6 48	1.4273	0.00108	.....
Mesitylene	$C_6H_3{\cdot}(CH_3)_2$	120 14	164	166	...	.....	.....	0.868	7.23	.....	.....	.....
Tetrahydronaphthalene	$C_{10}H_{12}$ ("Tetralin")	132.09	196	217	−12 5	80	<1	0.972	8.09	1.5461	0.00083	.....
Decahydronaphthalene	$C_{10}H_{18}$ ("Decalin")	138 14	186	194	.....	57	2	0 885	7.37	1.470	0.00088	.....

CHEMICAL PRODUCTS												
(*a*) *Sulfur Derivatives:*												
Carbon disulfide	CS_2	76 12	46	47	.....	−44	.....	1 270	10 58	1.620	. .....	95
Thiophene	$(CH{:}CH)_2S$	84.10	84	85	.....	.....	. ...	1.071	8.93	.....	.. ....	.....
Methyl sulfur cyanate	$CH_3 \cdot NCS$ (Methyl rhodanide)	73 10	133	135	.....	.....	.....	1 079	8 99	.....	.....	.....
Phenyl mustard oil	$C_6H_5 \cdot NCS$ (thiocarbanyl)	135.13	222	225	... .	.. ..	.. . .	1 135	9 46	.....	.....	.....
(*b*) *Chlorine Derivatives:*												
Dichlormethane	CH_2Cl_2	96 94	40	42	... ..	None	... ..	1 357	11 31	. .....	. .....	.....
S-Dichlorethylene	$CHCl{:}CHCl$	96.94	48	60	. ...	None	. ....	1.268	10 50	.....	..... .	.....
Chloroform	$CHCl_3$	119.38	60	62	.....	None	.....	1.490	12 42	1.4449	.... .	.....
Carbon tetrachloride	CCl_4	153 83	75	77	.. .	None	. ....	1 620	13.50	1.4607	. ... .	.....
Ethylene dichloride	$CH_2Cl \cdot CH_2Cl$ (S-dichlorethane)	98 95	80	86	−35 3	70	94	1 265	10 45	1 4446	0.00116	99
Trichlorethylene	$CHCl{:}CCl_2$	131 38	86	88	.. .	None	.....	1 473	12.28	1 4777	..	. ...
Iso-amyl chloride	$(CH_3)_2 \cdot CH \cdot CH_2 \cdot CH_2 \cdot Cl$	106 56	100	102	...	None	.....	0 880	7.33	. .....	..	....
β-Trichlorethane	$CH_2Cl \cdot CHCl_2$	133 40	114	117	....	None	.....	1.478	12 01	1.4709	.... .	.....
Epichlorhydrine	$CH_2{:}COH \cdot CH_2Cl$	92 51	117	119	.. .	None	.....	1 195	9 96	. .. .		.....
Tetrachlorethylene	$CCl_2{:}CCl_2$ (perchlorethylene)	165 83	115	122	. . .	None	.....	1.626	13.55	1 5050	. . .	.....
Monochlorbenzene	$C_6H_5 \cdot Cl$	112 50	130	132	−45 2	95	15	1.109	9.22	1.5250	0 00092	99
α-Monochlorhydrin	$CH_2OH \cdot CHOH \cdot CH_2Cl$	110 52	139	142	.. ..	None	.....	1.325	11 04	... .	....	.. .
S-Tetrachlorethane	$CHCl_2 \cdot CHCl_2$	167 85	144	147	... ..	None	.....	1.604	13 37	1 4509	. .. .	.....
Pentachlorethane	$CHCl_2 \cdot CCl_3$	202 30	159	161	... ..	None	.....	1.695	14 13	1 5020	. . .	....
Dichlorbenzene	$C_6H_4 \cdot Cl_2$	146 95	168	172	−17.6	175	3	1.308	10.88	1 549	0 00083	99
Dichlorethyl ether	$CH_2Cl \cdot CH_2O \cdot CH_2 \cdot CH_2Cl$	142.98	172	178	.. ...	185	2	1.222	10.17	1.457	0 00097	99
α-Dichlorhydrin	$CH_2Cl \cdot CHOH \cdot CH_2Cl$	128 97	174	180	.....	None	.....	1.375	11 46	....	. .	.....
Benzyl chloride	$C_6H_5 \cdot CH_2Cl$	126 53	176	180	.....	None	.....	1.105	9.21	.....	. . .	... .
Hexachlorethane	$CCl_3 \cdot CCl_3$	236 75	185	Subl.	.....	None	.....	2 090	17 42	.....	.. .	... .
Benzylidine chloride	$C_6H_5 \cdot CHCl_2$ (benzal chloride)	160 98	213	Subl.	..	None	..	1 295	10 80	. .....	.. .	.....
(*c*) *Nitrogen Derivatives:*												
Pyridine	$C_6H_5 \cdot N$	91 07	115	117	−100	...	.. .	0 988	8 23	.....	... .	.....
Aniline	$C_6H_5 \cdot NH_2$	93 08	180	183	−8	78	....	1.026	8 55	.....	..... .	.....
Phenylhydrazine	$C_6H_5 \cdot N \cdot H \cdot NH_2$	108 10	240	243	18	.....	.. ...	1 091	9 09	.....	.....	.....
Methyl cyanide	$CH_3 \cdot CN$ (acetonitrile)	41 04	82	83	... ..	.....	.....	0.783	6.53	. .....	.....	.....
Phenyl cyanide	$C_6H_5 \cdot CN$ (benzonitrile)	103.69	190	192	.. ...	. ...	.....	1 005	8.33	.....	.....	.....
Nitromethane	$CH_3 \cdot NO_2$	61.04	100	102	. ..	. ..	.....	1.144	9 54	.....	.....	.....
Nitrobenzene	$C_6H_5 \cdot NO_2$	123 07	215	218	5	90	.....	1.198	9.98	.. ..	.....	.....

* A.S.T.M. D268. † Pure material. ‡ A.S.T.M. D92. § Vapor pressures of mixtures cannot be determined by dynamic method. ‖ Very slightly soluble. ¶ Ester content appears under purity. ** Flash point below melting point. †† Sp. gr. determined with 14-inch hydrometer; accuracy, 0.0001.

TABLE XLIX—*Continued*

Name	Formula	Mol. Wt.	Distilling Range* Initial b. p. ° C.	Distilling Range* End point ° C	Melting Point† ° C.	Flash Point‡ ° C.	Vapor Pressure at 30° C. *Mm. Hg.*	Sp. Gr. at 20° C.††	Wt./Gal. at 20° C. Lb.	Refractive Index at 20° C.†	Expansion Coefficient 10–30° C.	Purity Commercial Product %
(*d*) *Aceto-Derivatives*												
Glacial acetic acid	$(CH_3 \cdot CO) \cdot OH$	60 04	117	118	15 5			1 050	8 75			99
Acetic anhydride	$(CH_3 \cdot CO)_2 \cdot O$	102 07	137	140				1 087	9 06			98
Cyclo-hexanolacetate	$CH_3 \cdot CO \cdot O \cdot C_6H_{11}$	142 14	170	175		64		0 970	8 09	1 4385		98
(*e*) *Alcohols:*												
Methanol (anhydrous)	CH_3OH	32 03	64	66	−97 8	52	160	0 793	6 60	1 329	0 00118	99
Ethanol (anhydrous)	C_2H_5OH	46 05	77	79	−117 3	65	78	0 791	6 58	1.361	0 00108	99
Isopropanol (anhydrous)	$(CH_3)_2CHOH$	60 06	82	83	−85 8	67	60	0 786	6 54	1.3776	0 00107	99
sec-Butanol	$CH_3CH_2CHOHCH_3$	74 08	96	103		88	26	0 808	6 72	1 397	0 00091	99
Isobutanol	$(CH_3)_2CH_2CHOH$	74 08	107	111	−108	111	17	0 803	6 68	1 396	0 00095	99
n-Butanol	C_4H_9OH	74 08	116	119	−89 8	111	11	0 811	6 75	1 3974	0 00094	99
sec-Amyl alcohol	$(CH_3CH_2)_2CHOH$	88 09	117	124		110	12	0 810	6 74		0 00088	95
Amyl alcohol (mixed isomers)	$C_5H_{11}OH$	88 09	121	139		111	§	0 814	6 77		0 00093	99
Methyl amyl alcohol	$(CH_3)_2CHCH_2CH(CH_3)OH$	102 11	131	132		114	6	0 807	6 72	1 4087	0 00100	99
Hexyl (2-ethyl butyl) alcohol	$CH_3CH_2CH(C_2H_5)CH_2OH$	102 11	144	156		145	4	0 833	6 93	1 4229	0 00089	99
Octyl (2-ethyl hexyl) alcohol	$C_4H_9CH(C_2H_5)CH_2OH$	130 14	182	201		178	<1	0 834	6 94	1 4300	0 00088	99
Cyclohexanol	$C_6H_{11}OH$	100 08	150	182	23 9	170	2	0 951	7 92	1 4656 (22 6° C.)	0 00077	94
Benzyl alcohol	$C_6H_5CH_2OH$	108 06	199	204	−15 3	212	<1	1 047	8 71	1 5399	0 00073	99
(*f*) *Ethers:*												
Ethyl ether	$(C_2H_5)_2O$	74 10	34	35		−3 5		0 720	6 00	1 351 (22° C)		99
Phenetol (ethyl phenyl)	$C_6H_5 \cdot O \cdot C_2H_5$	122 12	172					0 971	8 09			
Anisol (methyl phenylester)	$C_6H_5 \cdot O \cdot CH_3$	108 10	154		−37 8			0 999	8 33			
Methylcellosolve	$CH_3OCH_2CH_2OH$	76 06	121	126		105	<2	0 966	8 04	1 4028	0 00094	99
Cellosolve	$C_2H_5OCH_2CH_2OH$	90 08	133	137		111	7	0 931	7 75	1 4080	0 00097	99
Isopropylcellosolve	$(CH_3)_2CHOCH_2CH_2OH$	104 09	140	143		125	8	0 906	7 53		0 00093	97
Butylcellosolve	$C_4H_9OCH_2CH_2OH$	118 11	163	172		165	2	0 902	7 50	1 4190 (25° C)	0 00087	99
Methylcarbitol	$CH_3OCH_2CH_2OCH_2CH_2OH$	120 09	190	194		200	<1	1 035	8 62	1 4244 (26° C.)	0 00087	
Carbitol	$C_2H_5OCH_2CH_2OCH_2CH_2OH$	134 11	189	203		205	<1	1 027	8 55	1 3274	0 00082	

Butylcarbitol	$C_4H_9OCH_2CH_2OCH_2CH_2OH$	162.14	220	231		230	<1	0.955	7.95	1.4290 (27° C.)	0.00087	96
Benzylcellosolve	$C_6H_5CH_2OCH_2CH_2OH$	152.09	254	258		265	<1	1.070	8.90		0.00076	99
Diethylcellosolve	$C_2H_5OCH_2CH_2OC_2H_5$	118.11	119	125		90	22	0.849	7.07		0.00120	95
Diethylcarbitol	$C_2H_5OCH_2CH_2OCH_2CH_2OC_2H_5$	162.14	181	189		170	1	0.909	7.56		0.00106	99
(*g*) *Esters:* ¶												
Methyl acetate	CH_3COOCH_3	74.05	53	55	−98.1	5	263	0.908	7.55	1.3619	0.00134	82
Ethyl acetate	$CH_3COOC_2H_5$	88.06	70	80	−83.6	35	121	0.886	7.37	1.3727	0.00134	85
Isopropyl acetate	$CH_3COOCH(CH_3)_2$	102.08	84	94	−73.4	35	74	0.870	7.23	1.3770	0.00132	95
Isopropyl acetate	$CH_3COOCH(CH_3)_2$	102.08	82	90		45		0.857	7.13		0.00119	85
Ethyl propionate	$CH_3CH_2COOC_2H_5$	102.08	90	118	−72.6	70	43	0.891	7.41	1.3844	0.00125	96
sec-Butyl acetate	$CH_3COOCH(CH_3)(C_2H_5)$	116.09	105	127		88	29	0.858	7.14	1.389	0.00118	82
Isobutyl acetate	$CH_3COOCH_2CH(CH_3)_2$	116.09	114	118	−98.9	88	27	0.870	7.24	1.3997	0.00119	96
n-Butyl acetate	$CH_3COOC_4H_9$	116.09	119	127	−76.8	88	16	0.876	7.29	1.3951	0.00117	90
Ethyl butyrate	$CH_3CH_2CH_2COOC_2H_5$	116.09	107	131	−93.3	85	22	0.880	7.32	1.3932	0.00116	99
sec-Amyl acetate	$CH_3COOCH(CH_3)(C_3H_7)$	130.11	121	144		111	10	0.862	7.18		0.00113	92
Amyl acetate (mixed isomers)	$CH_3COOC_5H_{11}$	130.11	127	155		111	9	0.862	7.17		0.00104	84
Butyl butyrate	$CH_3CH_2CH_2COOC_4H_9$	144.12	152	170		128	4	0.874	7.27	1.4049	0.00107	99
n-Butyl propionate	$CH_3CH_2COOC_4H_9$	130.11	124	171		105	7	0.874	7.27		0.00106	99
Methyl amyl acetate	$CH_3COOCH(CH_3)CH_2CH(CH_3)_2$	144.12	140	147		113	2	0.857	7.13	1.4008	0.00109	95
Hexyl(2-ethyl butyl) acetate	$CH_3COOCH_2CH(C_2H_5)CH_2CH_3$	144.12	157	164		135	5	0.876	7.29	1.4103	0.00106	91
sec-Hexyl acetate	$CH_3COOCH(CH_3)(C_4H_9)$	144.12	129	158		110	10	0.861	7.16		0.00104	98
Ethyl lactate	$CH_3CH(OH)COOC_2H_5$	118.08	119	176		140	5	1.034	8.60	1.4118	0.00098	98
Octyl (2-ethyl hexyl) acetate	$CH_3COOCH_2CH(C_2H_5)C_4H_9$	172.16	195	203		180	<1	0.873	7.27	1.4300	0.00099	99
Cyclohexanyl acetate	$CH_3COOCH:(CH_2)_5$	142.11	165	193		165	7	0.963	8.01		0.00095	79
Isopropyl lactate	$CH_3CH(OH)COOCH(CH_3)_2$	132.09	149	167		130	5	0.988	8.22		0.00101	99
Butyl lactate	$CH_3CH(OH)COOC_4H_9$	146.11	145	230		176	5	0.980	8.16		0.00099	97
Glycol diacetate	$CH_3COOCH_2CH_2OOCCH_3$	146.08	184	191	−31	205	<1	1.107	9.21	1.415	0.00102	99
Diglycol diacetate	$CH_3COOCH_2CH_2OCH_2CH_2OOCCH_3$	190.11	238	251	−43	290	<1	1.116	9.29		0.00095	99
Diethyl carbonate	$(C_2H_5)_2CO_3$	118.08	87	127		85	17	0.957	7.96	1.3852	0.00113	91
3-Methoxy butyl acetate	$CH_3OCH(CH_3)CH_2CH_2OOCCH_3$	146.11	135	173		170	3	0.956	8.37		0.00100	99
Methylcellosolve acetate	$CH_3OCH_2CH_2OOCCH_3$	118.08	143	145		132	6	1.005	8.37	1.4025	0.00110	99
Cellosolve acetate	$C_2H_5OCH_2CH_2OOCCH_3$	132.09	145	166		122	3	0.974	8.10	1.4030 (25° C.)	0.00111	96
Butylcellosolve acetate	$C_4H_9OCH_2CH_2OOCCH_3$	160.12	188	192		180	<1	0.943	7.85		0.00104	99
Methylcarbitol acetate	$CH_3OCH_2CH_2OCH_2CH_2OOCCH_3$	162.11	203	212		180	<1	1.010	8.65		0.00099	99

* A.S.T.M. D268. † Pure material. ‡ A.S.T.M. D92. § Vapor pressures of mixtures cannot be determined by dynamic method. ‖ Very slightly soluble appears under purity. ¶ Ester content ** Flash point below melting point. †† Sp. gr. determined with 14-inch hydrometer; accuracy, 0.0001.

TABLE XLIX—*Continued*

Name	Formula	Mol. Wt.	Distilling Range* Initial b. p. ° C.	Distilling Range* End point ° C.	Melting Point† ° C.	Flash Point‡ ° C.	Vapor Pressure at 30° C. *Mm. Hg.*	Sp. Gr. at 20° C.††	Wt./Gal. at 20° C. Lb.	Refractive Index at 20° C.†	Expansion Coefficient, 10–30° C.	Purity Commercial Product %
Carbitol acetate	$C_2H_5OCH_2CH_2OCH_2CH_2OOCCH_3$	176.12	211	220	.....	225	<1	1.011	8.41	1.4230	0.00101	97
Butylcarbitolacetate	$C_4H_9OCH_2CH_2OCH_2CH_2OOCCH_3$	204.16	236	249	.....	260	<1	0.987	8.21	.....	0 00094	99
Methylal	$CH_2(OCH_3)_2$	76.07	42	44	.....	.....	.....	0.860	7.17	.....	.....	.....
Diethyloxalate	$(C_2H_5CO\cdot O)_2$	146.11	185	186	.....	.....	.....	1.085	9 04	.....	.....	.....
Acetyl acetone	$CH_3\cdot CO\cdot CH_2\cdot CO\cdot CH_3$	100.09	137	139	....	.....	.. ..	0 980	8.17	.....	.... ..	.....
(*h*) *Ketones:*												
Acetone	CH_3COCH_3	58.05	55	57	−94.3	0	277	0.793	6.60	1.3591	0.00134	99
Methylacetone	48% acetone–28% Me acetate–24% MeOH	...	54	70	.. ..	23	§	0.831	6 92	.....	0.00138	Mixt.
Methyl ethyl ketone	$CH_3COC_2H_5$	72.06	77	82	−86.4	34	119	0.809	6.73	1.3791	0.00128	99
Diethyl ketone	$C_2H_5COC_2H_5$	86 08	100	104	−42	59	46	0.815	6.78	1.3927	0.00113	99
Methyl propyl ketone	$CH_3COC_3H_7$	86.08	101	107	−77.8	60	48	0.810	6.74	1.3895	0.00111	99
Hexone (methyl isobutyl ketone)	$(CH_3)_2CHCH_2COCH_3$	100.09	112	118	−84.7	74	25	0.802	6.67	1.3959	0.00112	99
Methyl *n*-butyl ketone	$CH_3COC_4H_9$	100.09	114	137	−56.9	95	17	0 818	6 80	1.4024	0.00099	87
Mesityl oxide	$(CH_3)_2C{:}CHCOCH_3$	98 08	117	139	−59	90	<10	0.855	7.12	1.4439	0 00108	86
Diacetone alcohol	$(CH_3)_2C(OH)CH_2COCH_3$	116 09	153	160	. .	135	2	0.936	7.79	1.4204	0 00094	99
Dipropyl ketone	$C_3H_7COC_3H_7$	114.11	138	144	−32.6	120	9	0.816	6.79	1.4072	0.00107	99
Methyl *n*-amyl ketone	$CH_3COC_5H_{11}$	114.11	147	153	.. ..	115	7	0.818	6.81	1.4110	0.00103	98
Diisobutyl ketone	$(CH_3)_2CHCH_2COCH_2CH(CH_3)_2$	142.14	164	169	.....	145	4	0 808	6.72	.....	0.00102	95
Methyl *n*-hexyl ketone	$CH_3COC_6H_{13}$	128 12	169	173	−21.6	160	3	0 820	6 82	1.4161	0 00092	96
Cyclohexanone	$(CH_2)_5CO$	98 08	130	173	.. ...	145	7	0.946	7.87	1.4524 (16.8° C.)	0.00091	67
Methylcyclohexanone	$(CH_2)_4CH(CH_3){:}CO$	112 09	114	173	.....	145	4	0.921	7.67	.....	0 00085	74
Acetonylacetone	$CH_3COCH_2CH_2COCH_3$	114 08	188	193	−9	190	<2	0.973	8.10	1.449	0 00093	95
Phorone	$(CH_3)_2{:}C\cdot CHCOCH{:}C{:}(CH_3)_2$	138.11	114	198	28	180	<1	Crystals	.....	1.4998	.... ..	.....
Benzaldehyde	$C_6H_5\cdot COH$	106 08	177	179	.....	. ..	....	1.047	8.73	.....	.....	.....
Enanthaldehyde	$CH_3\cdot(CH_2)_5\cdot CO$ (enanthol)	113 13	155	157	.....	.....	.....	0.827	6.90	....	.....	.....
Diethylphthalate	$C_6H_4\cdot(CO\cdot O\cdot C_2H_5)_2$	222 17	290	300	.	140	.	1 125	9 38	1 500 (25° C.)	.....	.....

* A.S.T.M. D268. † Pure material. ‡ A.S.T.M. D92 § Vapor pressures of mixtures cannot be determined by dynamic method. ‖ Very slightly soluble. ¶ Ester content appears under purity. ** Flash point below melting point. †† Sp. gr. determined with 14-inch hydrometer; accuracy, 0.0001.

face-tension at 20° C. expressed in dynes per cm. in respect to their effect upon asphalts:

1. Flocculating solvents:	
Ethyl ether	17.1 dynes per cm.
Petroleum naphtha (b. pt. 40 to 60° C.)	17.4
Normal hexane	17.4
Ethyl acetate	22.9
Acetone	23.1
2. Solvents combining both flocculating and peptizing properties:	
Carbon tetrachloride	25.7 dynes per cm.
Cyclo-hexane	26.0
3. Peptizing solvents:	
Chloroform	27.1 dynes per cm.
Toluol	28.4
Benzol	28.86
Ortho-xylol	29.9
Carbon disulfide	31.3
Pyridine	35.5
Benzyl acetate	36.0
Furfurol	40.7
Aniline	42.6
Nitrobenzol	43.4

It has been shown [3] that asphalt is soluble in a mixture of two solvents of the correct surface-tension, when it is insoluble of either component separately. Thus, a mixture of equal parts of ethyl ether and aniline, or a mixture of equal parts of petroleum naphtha and benzyl acetate, will completely dissolve asphalt which would be flocculated by ethyl ether or petroleum naphtha when used alone.

The surface-tension of asphalts at elevated temperatures shows but little variation among different specimens. Although their surface-tension at 20° C. cannot be directly measured, they must certainly be higher than 30 dynes per cm., and hence must be within the range of the peptizing solvents. Direct measurements [4] show that an asphalt showed the following surface tensions at varying temperatures:

0° C.	33.6 dynes per cm.
25° C.	32.3 " " "
50° C.	30.8 " " "
75° C.	29.3 " " "
100° C.	27.6 " " "

Coal tars and coal-tar pitches show a considerably higher surface-tension than do asphalts (i. e. about 8 dynes per cm. higher), hence solvents which act as peptizing agents for asphalts, may behave as flocculents for coal-tar products. Their high surface-ten-

TABLE L

Solvents Derived from Coal Tars

Name	Color	Sp. gr. at 15½° C.	Distillation				A.S.T.M. Closed Cup Flash Point	Solidifying Point
			First Drop	Per Cent at Fixed Temperature	Dry Point	Range		
Nitration benzol	Water wh.	0 8820–0 8860	Includes 80 2° C.		.....	1° C.	10–20° F.	4 75° C.+
Non-corrosive commercially pure benzol	" "	0.875–0.886	Includes 80 2° C.		.....	2° C.	10–20° F.	±41° F. ± 5° C.
Commercially pure benzol	" "	0 875–0.886	Includes 80 2° C.		.....	2° C.	10–20° F.	±41° F. ± 5° C.
Non-corrosive 90 per cent benzol	" "	0 870–0 882	78 2° C.+	90–95% at 100° C.	120° C.–	42° C.	10–20° F.	±36° F. ±2.5° C.
Commercial 90 per cent benzol	" "	0 870–0 882	76 2° C.+	90–95% at 100° C.	120° C.–	44° C.	10–20° F.	±36° F. ±2 5° C.
Motor benzol	" "	....	176–180° F. / 80– 82° C.	75% + at 100° C.	284° F. / 140° C.	± 60° C.	10–20° F.	±36° F. ±2 5° C.
Motor benzol	" "	0 860–0.880	168–180° F. / 76– 82° C.	60% at 100° C. / 90% at 120° C.	338° F. / 170° C.–	± 94° C.	10–20° F.	±36° F. ±2 5° C.
Nitration toluol	" "	0 869–0 873	Includes 110 4° C.		.....	1° C.	±55° F.	.....
Non-corrosive commercially pure toluol	" "	0 864–0 874	Includes 110 4° C.		.....	2° C.	±55° F.	.....
Commercially pure toluol	" "	0 864–0 874	Includes 110 4° C.		.....	2° C.	±55° F.	.....
Commercial toluol	" "	0 860–0 870	±98° C.	90% at 120° C.	±132° C.	±34° C.	±40° F.	.....

Special commercial xylol	" "	0.860–0 875	±122° C.	5%—at 130° C.	150° C.—	±28° C.	75° C.+	.. ..
3° xylol	" "	0.865–0 870	137.2° C.	... .	140 5° C.	3° C.	75–80° F.	.....
5° xylol	" "	0 860–0 870	137° C.+	....	142° C.	5° C.	75–80° F.	.....
10° xylol	" "	0 860–0 870	135° C.	....	145° C.	10° C.	75–80° F.	.. ..
Nitration meta xylene	" "	0.865–0 870	138 3° C.	Includes 139 5° C.	139 7° C.	1 5° C.	75–80° F.	.. ..
Solvent naphtha	" "	0 860–0 875	±125° C.	5%—at 130° C. 90–95% at 160° C.	180° C.	±55° C.	75° F.+	.....
Special washed solvent naphtha	" "	0.860–0 875	±125° C.	5%—at 130° C. 90–95% at 160° C.	180° C.—	±55° C.	75° F.+	.....
Special solvent naphtha	" "	0 820–0 850	±140° C.	... .	±185° C.	45° C.	100° F.+	.....
Crude light solvent naphtha	Light amber to dark red	0 860–0 875	±125° C.	10%—at 130° C. 90%+at 160° C.	±200° C.	±75° C.	75° F.+	.....
Straw color solvent naphtha	Light straw color	0 865–0 895	±125° C.	....	±165° C.	±40° C.	75° F.+	.....
Cumene	Water wh.	0 860–0.880	165.5° C.	50% from 160° C.—171° C.	175.5° C.	±10° C.	110° F.+	.....
Low boiling hiflash naphtha	" "	0 860–0 890	160° C.	....	185° C.	25° C.	100° F.+	.....
Hiflash naphtha	" "	0 860–0.890	150° C.	90% at ±180° C.	200° C.	50° C.	100° F.+	.....
High boiling hiflash naphtha	" "	0.860–0.890	175° C.	.....	200° C.	25° C.	100° F.+	.....
Heavy solvent naphtha	Amber	0.930–0 980	±160° C.	.....	200° C.	±65° C.	100° F.+	No $C_{10}H_8$ crystals at −20° C.
Heavy solvent naphtha	Amber to red	0.870–0.890	±150° C.	10%—at 160 °C. 90%+at 200° C.	215° C.	±65° C.	100° F.+	"
Heavy solvent naphtha	Light amber to dark red	0.925–0 950	±150° C.	10%—at 160° C. 70% at 200° C. 95%+at 260° C.	285° C.	±130° C.	100° F.+	"
Heavy solvent naphtha	Light amber to dark	0 940–0 986	±155° C.	5% —at 160° C. 90%+at 200° C.	285° C.	±130° C.	100° F.+	"
Crude heavy solvent naphtha	Light amber to dark red	0.930–0.986	+115° C.	5%—at 120° C. 90%+at 200° C.	220° C.	±105° C.	±40° F.	.....

sion favors the peptization of the carbon ultramicrons, hence much coarser carbon may be kept in solution or suspension. Peptizing agents for coal-tar products include pyridine, furfurol, ortho-nitro-toluol, aniline and nitrobenzol.

It has been observed that in the case of certain bituminous substances, e.g. coal tars and coal-tar pitches, various solvents are adsorbed by the bituminous constituents, resulting in the flocculation of insoluble compounds, which form in gradually increasing quantities over a considerable period of time. With benzol or toluol for example, the filtrate initially appears perfectly clear under an ordinary microscope, and even under an ultra-microscope shows no evidence of particles having Brownian movement; whereas upon standing at room temperature, a gradually increasing formation of precipitate is noted under an ordinary microscope. On the other hand, the filtrate obtained from a solution of coal tar or coal-tar pitch in aniline remains clear when examined under an ordinary microscope, both initially and upon standing, whereas under an ultra-microscope it forthwith exhibits a rich field of particles having Brownian movement. If benzol or toluol is added to the aniline solution, the particles are observed to lose their mobility under the ultra-microscope and in turn become visible under an ordinary microscope. Solutions of coal tar or coal-tar pitch in selenium oxychloride (which is the best known solvent for these products) when filtered through an alundum thimble, appear optically clear when examined under the ultra-microscope, both initially and upon standing, without any evidence of Brownian movement, thereby showing that selenium oxychloride has neither a peptizing nor a flocculating effect.

Every solvent, therefore, must be studied separately, since each behaves differently, not only in relation to other solvents, but also with respect to different bituminous substances, or even with various grades of the same generic substance.[5]

In this connection, it is interesting to note that a method has been proposed for separating asphalt and coal-tar pitch mixtures by treatment with a solution consisting of 2 volumes of carbon disulfide to 1 volume of petroleum naphtha (40 to 60° C. distillation range), boiling, cooling and filtering off the precipitated tar colloid. The extract is then evaporated and the residue repeatedly extracted with a cold mixture of 1 volume aniline to 1 volume of 96 per cent

TABLE LI.—Proximate Solubility at Room Temperature of Average Commercial Bituminous Substances (¼ gm. plus 10 ml. Solvent; 24 hrs.) Exclusive of Mineral Constituents and Free Carbon.

Legend: a = Completely Soluble (>90%); b = Mostly Soluble (90 to 50%); c = Partly Soluble (50 to 25%); d = Slightly Soluble (25 to 10%) e = Insoluble (<10%).

Genus	Species	Member	Hydrocarbons						Sulfur Derivatives				Chlorine Derivatives													Nitrogen Derivatives						Aceto Derivatives			Alcohols							Ethers, Esters, Ketones, Aldehydes, etc.								
			Petroleum Naphtha	Turpentine	Benzol	Cumol	Mesitylene	Tetralin	Carbon Disulfide	Thiophene	Methyl Sulfocyanate	Phenyl Mustard Oil	Chloroform	Carbon Tetrachloride	o-Dichlorethylene	Ethylene Dichloride	Trichlorethylene	Tetrachlorethylene	Pentachlorethane	Iso-amyl chloride	Monochlorhydrin	α-Dichlorhydrin	Epichlorhydrin	Monochlorbenzene	Benzyl-chloride	Benzal-chloride	Pyridine	Aniline	Phenylhydrazine	Methylcyanide	Benzo-nitrile	Nitromethane	Nitrobenzene	Glacial-acetic Acid	Acetic anhydride	Cyclohexanyl acetate	Methyl alcohol	Ethyl alcohol	Allyl alcohol	Iso-amyl alcohol	Amylene hydrate	Hexalin	Benzyl alcohol	Ethyl ether	Methylal	Acetone	Diethyl oxalate	Diacetone alcohol	Acetyl acetone	Enanthol	Phenetol	Anisol	Benzaldehyde	Diethyl-phthalate
Bitumens	Natural Waxes	Ozokerite	de	cd	bc	bc	cd	cd	bc	cd	de	bc	b	cd	ab	de	d	ab	de	de	ab	de	de	de	de	de	de	e	e	e	e	e	e	e	de	de	de	de	de	de	de	de	de	de	de	de	de	de	de	de	de
		Ceresine (commercial)	c	c	a	a	a	c	a	c	de	bc	a	c	a	de	de	de	de	d	e	e	e	e	d	d	de	e	e	e	e	e	e	d	e	e	e	de	de	de	de	de	de	de	de	de	de	de	d	d	d	d
		Montan wax	cd	cd	cd	bc	cd	de	ce	de	de	cd	cd	cd	c	cd	e	de	de	de	e	e	e	d	a	d	de	de	e	e	e	e	e	e	e	de	de	de	de	e	de	de	e	de	de	de	de	de	de	de	de	de
	Natural Asphalts	<10% mineral matter (maltha)	b	a	a	a	a	a	a	ab	cd	a	a	a	a	a	ab	a	a	ab	bc	bc	b	a	ab	a	a	d	cd	bc	b	cd	a	d	d	a	de	de	de	de	cd	c	bc	d	b	cd	d	de	de	de	cd	ab	b	bc	b	d
		>10% " " (e.g. Trinidad)	cd	ab	ab	b	b	ab	a	ab	de	a	a	ab	b	a	ab	b	a	ab	bc	b	e	c	b	b	bc	ab	ab	de	de	de	bc	de	e	e	de	de	de	de	cd	c	cd	b	cd	de	de	de	de	d	c	bc	c	de
	Asphaltites	Gilsonite	c	bc	ab	c	bc	c	a	a	cd	ab	a	a	ab	b	c	c	b	cd	bc	e	c	de	a	ab	bc	a	bc	de	d	e	c	de	bc	de	e	e	e	e	e	de	de	cd	c	de	cd	e	de	e	e	c	c	cd	cd	c	de
		Glance pitch (Syrian, Cuban, etc.)	cd	bc	a	b	b	ab	a	ab	cd	a	a	a	ab	ab	cd	cd	cd	b	e	c	de	a	ab	ab	ab	b	de	de	e	e	bc	e	de	e	e	e	e	de	de	cd	b	bc	bc	b	de	d	e	e	e	e	c	b	de
		Grahamite	de	d	ab	bc	cd	c	a	ab	de	ab	bc	cd	cd	cd	de	de	de	d	e	de	e	bc	cd	bc	cd	e	e	e	e	cd	e	bc	d	e	e	e	e	e	e	e	e	d	e	d	e	de	e	e	e	e	d	e	e
Pyrogenous Distillates	Pyrog. waxes	Paraffin wax	a	a	a	b	a	c	a	d	e	a	a	a	c	de	c	ab	de	d	e	e	e	c	d	d	d	d	de	e	e	e	d	e	e	e	e	e	de	e	e	e	e	de	de	e	de	de	e	e	d	d	e	de
	Petroleum Tars	Oil-gas tar	d	c	ab	a	c	b	b	a	a	a	a	a	c	c	bc	b	a	c	b	a	a	ab	a	a	a	a	ab	de	a	c	a	c	c	c	de	de	c	de	de	c	a	c	c	c	bc	c	c	c	b	c	a	ab
	Coal Tars	Gas-retort coal-tar	cd	b	ab	ab	b	c	ab	ab	ab	a	a	a	c	c	b	ab	a	b	b	b	b	ab	bc	a	b	ab	ab	a	c	b	de	ab	c	c	c	d	d	c	cd	de	c	c	e	bc	bc	e	b	cd	b	ab	
		Low-temperature coal-tar	de	c	a	a	b	b	ab	b	bc	a	ab	b	c	c	ab	ab	b	cd	b	c	de	b	b	ab	c	de	b	a	bc	de	bc	c	cd	c	cd	d	de	de	de	a	c	c	de	c	de	b	b	ab	de	de	b	
	Wood Tars	Pine tar	cd	a	a	a	bc	c	ab	a	a	ab	a	bc	a	a	a	a	cd	b	a	a	a	a	a	d	a	a	a	b	a	c	a	d	d	d	a	a	a	a	a	a	a	a	a	a	a	a	a	a	a	a	a	a
		Hardwood tar (beech tar)	de	c	a	ab	a	b	bc	a	a	ab	a	cd	a	a	a	ab	d	b	c	b	a	a	a	ab	b	bc	a	a	b	a	a	c	a	a	a	a	a	a	a	a	a	c	a	a	d	a	ab	a	a	a	a	a	a
	Miscellaneous Tars	Peat tar	cd	c	a	ab	a	a	bc	b	b	a	bc	a	c	c	b	c	ab	b	b	d	a	a	c	a	c	d	ab	d	d	c	cd	a	d	d	d	c	c	c	d	c	bc	c	c	c	bc	d	b	a	b	b	bc	
		Lignite tar	bc	a	a	ab	a	a	a	a	c	a	a	a	a	c	ab	b	a	a	bc	b	a	a	c	b	a	a	a	c	a	b	e	a	c	d	d	c	de	c	a	ab	b	a	a	a	bc	a	a	b	a	a	ab	a	c
		Shale tar	c	c	a	ab	a	bc	b	c	c	a	a	a	a	c	b	b	c	b	b	c	c	a	a	c	b	a	a	c	a	b	e	a	c	d	d	c	de	c	b	de	c	c	c	c	c	bc	d	b	a	b	b	c	
		Bone tar	bc	b	a	b	ab	a	a	b	b	a	a	a	a	c	b	ab	a	b	b	a	a	ab	a	a	a	a	a	e	a	c	a	a	c	d	d	c	ab	c	a	ab	b	ab	a	a	ab	a	a	b	a	a	ab	a	c
Pyrogenous Residues	Pyrogenous Asphalts	Residual oils	b	a	a	a	a	a	a	a	bc	a	a	a	a	a	ab	a	a	a	bc	c	bc	a	a	a	a	a	a	d	cd	c	b	d	a	d	d	b	de	de	de	d	d	a	b	bc	ab	cd						
		Blown petroleum asphalt	bc	ab	a	a	ab	a	a	a	cd	a	a	a	ab	a	bc	b	bc	ab	cd	d	c	a	a	a	ab	a	de	cd	de	cd	b	de	a	d	d	c	de	de	de	d	a	bc	c	b	cd	d						
		Residual asphalt (soft)	bc	ab	a	ab	ab	ab	a	a	cd	a	a	a	b	a	bc	bc	bc	de	d	d	a	a	a	ab	bc	de	de	cd	de	de	c	e	a	d	d	c	de	de	de	d	a	ab	c	c	b	de						
		Residual asphalt (hard)	cd	bc	a	b	b	bc	a	a	de	ab	a	a	bc	a	cd	e	c	bc	e	de	de	a	a	ab	b	b	bc	de	de	e	e	c	e	ab	e	e	e	de	d	de	cd	cd	c	cd	cd	de	cd	c	cd	b	d	d	bc	e
		Sludge asphalt	cd	bc	ab	b	bc	c	ab	ab	de	a	ab	ab	c	b	d	cd	cd	bc	e	de	de	ab	b	b	ab	cd	e	de	e	de	c	e	ab	de	e	e	de	d	de	cd	cd	c	cd	cd	de	cd	e	e				
	Petroleum Pitch	Oil-gas-tar pitch	de	c	ab	b	c	bc	b	ab	c	ab	a	ab	c	c	c	bc	a	cd	c	bc	b	b	b	a	b	a	b	de	b	cd	b	d	d	d	e	e	d	de	e	d	c	cd	d	c	cd	cd	d	cd	cd	b	bc	
	Coal-tar Pitch	Gas-works coal-tar pitch	de	c	ab	b	bc	cd	ab	ab	c	a	ab	ab	c	c	c	ab	ab	d	c	c	b	c	ab	b	ab	ab	ab	de	c	de	ab	de	de	d	de	de	c	cd	de	c	c	cd	d	c	c						
	Wood-tar Pitches	Pine-tar pitch	cd	ab	a	a	c	c	ab	ab	a	c	a	bc	ab	c	b	cd	a	cd	b	b	a	a	a	a	b	ab	ab	b	b	c	a	a	b	ab	ab	ab	ab	ab	b	ab	ab	ab	ab	ab	a	a	b	b	a	a	
		Hardwood-tar pitch (beech)	de	c	a	b	b	c	bc	ab	a	c	ab	cd	a	c	c	d	c	d	a	a	a	ab	bc	cd	ab	b	bc	c	a	d	c	ab	ab	ab	ab	a	cd	c	ab	ab	c	b	ab	ab	ab	a	a	c	b	a	a	
	Miscellaneous Pitches	Rosin pitch	bc	a	a	a	a	c	ab	a	a	a	a	bc	b	c	c	b	a	ab	b	cd	de	c	d	c	ab	a	a	b	c	b	b	d	c	d	de	e	e	b	b	ab	ab	d	b	b	b	ab	c	de				
		Peat-tar pitch	cd	ab	a	bc	ab	a	bc	b	de	a	bc	a	c	c	ab	b	b	cd	c	d	b	ab	c	b	ab	ab	cd	ab	de	e	c	d	ab	d	d	d	e	e	d	d	d	c	ab	d	d	b	d	de				
		Lignite-tar pitch	cd	bc	ab	c	bc	ab	a	ab	c	a	ab	ab	ab	c	c	ab	b	b	cd	e	c	d	a	ab	ab	ab	ab	a	cd	b	e	d	ab	d	d	d	de	de	d	de	d	ab	a	a	b	a	b	d	de			
		Bone-tar pitch	de	bc	a	c	bc	ab	a	b	cd	a	a	ab	ab	c	ab	c	c	a	ab	c	c	a	ab	c	bc	a	a	ab	d	a	c	cd	b	c	d	de	de	de	cd	a	c	cd	b	bc	d	de	de	de	d	de			
		Stearin pitch (soft)	ab	a	a	a	a	a	a	a	cd	a	ab	a	a	b	a	a	a	ab	cd	c	cd	a	ab	a	a	c	cd	de	bc	cd	cd	de	d	d	de	de	de	de	cd	ab	ab	d	b	cd	de	cd	de	de	de	cd	ab	b	a	ab	d	de
		Stearin pitch (medium and hard)	bc	b	a	a	ab	ab	a	a	cd	a	ab	a	a	bc	ab	a	a	ab	cd	cd	ab	ab	ab	ab	ab	cd	de	bc	bc	cd	de	de	d	de	de	de	de	de	bc	cd	de	de	cd	de	de	de	de	de	de	de	de	de	de
		Cotton pitch	c	a	a	a	ab	b	ab	a	de	a	a	ab	ab	bc	bc	ab	a	a	ab	cd	cd	ab	ab	ab	ab	c	b	cd	bc	bc	cd	de	d	de	de	de	de	de	bc	cd	de	de	cd	de	de	de	de	de	de	de	de	de	de
		Wool-grease pitch	a	bc	b	cd	bc	bc	ab	a	de	ab	cd	bc	bc	c	ab	a	ab	ab	de	de	de	c	cd	b	d	c	cd	de	de	de	de	d	de	de	de	de	de	de	cd	d	de	de	de	de	de	de	de	de	de	de	de	de
		Montan pitch	c	c	c	c	c	bc	a	c	c	a	c	b	a	cd	c	b	c	a	bc	e	d	d	cd	d	e	b	c	de	de	de	de	de	b	de	de	de	de	de	de	c	d	de	de	de	de	de	de	de	de	de	de	de	de
		Naphthol pitch	de	c	bc	cd	cd	d	bc	c	b	a	a	d	d	d	d	c	c	a	c	de	d	d	cd	d	e	b	a	ab	de	b	e	d	de	e	e	de	de	de	de	b	b	de	de	de	de	de	de	de	de	de	de	de	de

To face page 525.

ethyl alcohol, which dissolves out the tar medium, leaving the asphalt, which may be dried and weighed.[6] Similarly, a colorimetric method has been proposed,[7] which consists in dissolving the mixture in 10 parts of carbon disulfide, diluting with 90 parts of petroleum naphtha, shaking, and dropping upon a piece of filter paper, whereupon characteristic forms are produced, the colors of which aid in the identification of coal-tar products and asphalt respectively.

Solubility of Bituminous Substances. The extent to which typical bituminous substances dissolve in various solvents is roughly indicated in Table LI.[8] It should be noted that the results are only "proximate," and are subject to considerable variation depending upon the particular characteristics of the specimen taken for examination. In other words, a relatively small difference in the physical properties (e.g. hardness) of the substance (brought about by distillation, heating, aging, blowing, blending, or the like) will result in a marked difference in its solubility in the various menstra.

Adsorption of Bituminous Substances. Mineral fillers have the property of adsorbing bituminous substances in varying amounts, depending upon the particular characteristics of the former. This may be demonstrated by dissolving the bituminous substance in benzol and agitating the mixture with a weighed quantity of the finely-divided filler. The adsorption may be measured by the diminution of the depth of color of the solution. This phenomenon may also be indicated by boiling a mixture of the bituminous substance and filler with water, to which is successively added sodium carbonate in fractional molar proportions (i.e. 106 g. per liter) until a separation of the bituminous matter separated from the mineral constituents.[9] It has been claimed that fillers that are activated by heat are superior to those that are not so activated.[10]

CHAPTER XXVII

SOLID, SEMI-SOLID AND SEMI-LIQUID BITUMINOUS COMPOSITIONS

Adhesive Compounds for Built-up Roofing and Waterproofing Work. Tar-pitches and asphaltic products are used for this purpose, similar in composition to the surface coatings of sheet roofings. Adhesive compounds are employed in three classes of work, viz.:

(1) For constructing membrane waterproofings on underground work exposed to uniformly moderate temperature conditions, as for example tunnels, foundations of buildings, retaining walls, culverts, dams, conduits, tanks, etc.

(2) For constructing membrane waterproofings on structures above ground, exposed to wide fluctuations in temperature and subjected to severe vibration, as on bridges, culverts, etc.

(3) For constructing built-up roofs exposed to extremely wide fluctuations in temperature, and to little or no vibration.

Each class will be considered separately.

Adhesive Compounds for Damp-proofing and Waterproofing:

(*a*) Below Ground. Since underground structures are exposed to uniformly moderate temperature conditions (e.g. not exceeding 115° F.), the adhesive compositions are usually prepared of a comparatively low fusing-point. The two most important requirements are that they should have great ductility and a high tensile-strength.

(*b*) Above Ground. Bituminous materials used under these conditions must necessarily withstand wide variations in atmospheric temperature without softening or becoming brittle; in other words they should possess a low susceptibility index.

Specifications for asphalt and coal-tar pitch intended for damp-proofing and waterproofing purposes are given in Tables LII and LIII, respectively.

TABLE LII

ASPHALT FOR DAMP-PROOFING AND WATERPROOFING

Test No.	Description of Test	Alternate No. 1		Alternate No. 2
		For Use Below Ground *	For Use Above Ground	For Use Both Below and Above Ground
		A.S.T.M. [1]	A.S.T.M. [2]	F.S.B. [3]
2*a*	General appearance	Homogeneous	Homogeneous	.....
3	Appearance surface after 7 days	.....	.....	Bright and glossy
9*b*	Penetration at 115° F. ..... Max.	.....	100	100
	Penetration at 77° F. ..... Range	50–100	25–50	25–50
	Penetration at 32° F. ..... Min.	.....	10	10
10*a*	Ductility at 77° F., cm. ..... Min.	30	15	15
15*b*	Fusing-point (R. and B.), ° F. .. Range	115–145	150–170	140–171
16	Loss at 325° F., 5 hrs., per cent. . Max.	2	1	1
	Per cent orig. pen. of resid. at 77° F. ..... Min.	60	60	60
17*b*	Flash-point (open-cup), ° F. .... Min.	347	400	.....
21	Insoluble in carbon disulfide, per cent. ..... Max.	5	1	1 †
25	Water, per cent. ..... Max.	0	0	.....

* For use at exposed temperatures not exceeding 115° F.
† Shall be mainly silica or silicates.

Adhesive Compounds for Built-up Roofing Work. This class may either consist of coal-tar [4] or asphaltic products and they are used in structures where there is little to no vibration. The same materials are adapted for this purpose as for the coating compounds of prepared roofings, but they are generally prepared of a softer consistency and lower fusing-point. The susceptibility index does not play an important rôle, and the ductility may be considerably less than in the foregoing class of adhesives. Specifications for asphalt and coal-tar pitch intended for built-up roofing purposes are included in Tables LIV and LV, respectively.

In Germany, a compound is commonly used in the construction of built-up roofs, known as "Holzzement," prepared by heating coal-tar pitch with sulphur at 250° F. As this treatment reduces the adhesiveness of the pitch, it is often customary to add a proportion of resin or rosin-oil to the sulfurized product to remedy this defect.[5] The addition of a small percentage of aliphatic or aro-

matic hydrocarbons or terpenes is claimed to accomplish the same results;[6] or trichlorethylene in the case of asphalts; likewise pyridine in the case of coal-tar pitch.[7]

TABLE LIII

COAL-TAR PITCH FOR DAMP-PROOFING AND WATERPROOFING

Test No.	Description of Test	Alternate No. 1				Alternate No. 2
		For Use Below Ground		For Use Above Ground *		For Use Below and Above Ground †
		High Carbon	Low Carbon	High Carbon	Low Carbon	High or Low Carbon
		A.S.T.M.[8]	A.S.T.M.[9]	A.S.T.M.[10]	A.S.T.M.[11]	F.S.B.[12]
2a	General appearance	Homog.	Homog.	Homog.	Homog.	.....
3	Appearance surface after 7 days	.....	.....	.....	.....	Bright and glossy
7	Specific gravity at 77° F. Range	1.24–1.34	1.21–1.30	1.25–1.35	1.22–1.31	1.22–1.34
10a	Ductility at 77° F., cm. Min.	50	50	20	20	50
15c	Fusing-point (Cube), ° F. Range	110–140‡	110–140‡	130–155‡	130–155‡	125–140
20a	Distillation test (per cent by weight):					
	Distillate below 572° F. Max.	12	15	10	12	12
	Sp. gr. ditto at 100°/77° F. Min.	1.03	1.03	1.03	1.05	1.03
	Residue at 572° F. Min.	88	85	90	88	.....
21	Insoluble in carbon disulfide, per cent. Range	20–35	10–25	22–37	12–27	15–30
21	Ash (on ignition), per cent. Max.	.....	.....	.....	.....	1.0
25	Water, per cent. Max.	0	0	0	0	.....

* For use at exposed temperatures not exceeding 115° F.
† For use at exposed temperatures not exceeding 100° F.
‡ Specified to have a range not exceeding 10° F. within the limits prescribed.

Plastic-slate Cement. This is used in the construction of built-up roofs, for the finishing off of flashings, parapet walls, around chimneys, vent-pipes, etc. It consists of a mixture of 20 to 25 parts by weight of refined coal tar from which the volatile oils have previously been removed by distillation, and 80 to 75 parts by weight of finely ground slate. Sometimes, powdered shale, marl, or loam is used as filling. These ingredients are mixed together while hot —usually in a small fire-heated kettle near where it is to be used, and the "plastic-slate" is trowelled in place before cooling. The mineral powder prevents the tar from running or checking on exposure, and at the same time materially increases its weather-resistance.

Calking Compounds. This class of materials includes sundry adhesives and cements, intended for use as fillers for expansion-

TABLE LIV

Asphalts for Built-up Roofs (Surfaced and Unsurfaced)

Test No.		Alternate No. 1			Alternate No. 2		
		Surfaced with Slag or Gravel	Finished with Asphalt Mopping Coat		Surfaced with Slag or Gravel	Finished with Asphalt Mopping Coat	
	Over boards.....Max. incline	3 in. per ft.	3 in. per ft.	6 in. per ft.	3 in. per ft.	3 in. per ft.	6 in. per ft.
	Over concrete of gypsum.....Max. incline	3 in. per ft.	3 in. per ft.	6 in. per ft.	1½ in. per ft.	1½ in. per ft.	3 in. per ft.
		A.S.T.M.[13]			F.S.B.[14]		
3	Appearance surface.....	.....	.....	.....	Bright	Bright	Bright
9*b*	Penetration at 115° F.....Max.	100	150	100	.....	100	100
	Penetration at 77° F.....Range	20–50	20–40	20–40	20–50	20–50	20–50
	Penetration at 32° F.....Min.	5	5	10	10	10	10
10*a*	Ductility at 77° F., cm.....Min.	10	4	3	5	5	5
15*b*	Fusing-point (R. and B.), ° F.....Range	135–150	135–170	170–200	140–165	150–165	165–190
16	Loss at 325° F., 5 hrs., per cent.....Max.	1.5	1.5	1.5	1.5	1.0	1.0
	Per cent orig. pen. of resid. at 77° F.....Min.	60	60	60	60	60	60
21	Soluble in carbon disulfide, per cent.....Min.	99* 65†	99* 65†	99* 65†	.....	.....	.....
22	Soluble in carbon tetrachloride, per cent....Min.	99*	99*	99*	.....	.....	.....
21	Ash on ignition, per cent.....Max.	1*	1*	1*	1*	1*	1*
	Ash on ignition, per cent.....Range	7.5–35*	7.5–35†	7.5–35†	10–35†	10–35†	10–35†
	Per cent of ash retained on:						
	200 mesh sieve.....Max.	15	15	15	.....	.....	.....
	100 mesh sieve.....Max.	.....	.....	.....	2†	2†	2†
25	Water, per cent.....Max.	0	0	0	.....	.....	.....

* Unfilled asphalts.
† Filled asphalts and native asphalts.

joints, cracks, holes or imperfections in masonry and cement construction; also for sealing the spaces between window- or door-frames and the adjoining masonry; likewise along cornices, copings, etc.; also for calking the wooden decks of ships, etc.[15] They are composed of suitable bituminous substance (e.g. natural or petroleum asphalt, or else coal-tar pitch) with inert fillers and asbestos fibers, and sometimes cut back with liquid tar, non-drying or treated drying oils, or volatile solvent to impart plasticity. Rubber, resins, or waxes may be incorporated to suitably modify the properties of the product. A colored calking cement has been proposed consisting of paracoumarone resin (melting-point 50–60° C.), asbestos fibers, colored pigment and a coal-tar solvent.[16] These are marketed in two grades, one in such a plastic state that it may be forced into the joint or crevice by means of a pressure gun, and the other of a stiffer consistency suitable for application with a knife, trowel, or other hand tool.[17]

TABLE LV

COAL-TAR PITCH FOR BUILT-UP ROOFS SURFACED WITH SLAG OR GRAVEL

Test No.		Alternate No. 1		Alternate No. 2
	Over boards.....Max. incline Over concrete or gypsum. Max. incline	3 in. per ft. 1 in. per ft.	3 in. per ft. 1 in. per ft.	3 in. per ft. 1½ in. per ft.
		High-carbon	Low-carbon	High or Low Carbon
		A.S.T.M.[18]	A.S.T.M.[19]	F.S.B.[20]
2*a*	General appearance.....	Homogeneous	Homogeneous	.....
3	Appearance surface after 7 days	.....	.....	Bright and glossy
7	Specific gravity at 77° F.....Range	1.25–1.34	1.22–1.32	1.22–1.34
10*a*	Ductility at 77° F., cm.....Min.	50	50	50
15*c*	Fusing-point (Cube), °F.....Range	140–150	140–150	140–150
20*a*	Distillation test (per cent by weight):			
	Distillate below 572° F.....Max.	12	12	12
	Sp. gr. ditto at 100°/77° F...Min.	1.03	1.03	1 03
	Residue at 572° F.....Min.	88	88	.....
21	Insoluble in carbon disulfide, per cent.....Range	22–35	15–22	15–30
	Mineral matter (by ignition), per cent.....Max.	0.5	0.5	1.0
25	Water, per cent.....Max.	0	0	.....

Bituminous Grout.[21] This is intended for use as a protective covering for membrane waterproofing, or as a bedding for bricks, or for filling the joints of bricks, or flooding the surface of bricks, in connection with railroad bridges, culverts, subways, tunnels, etc.[22] It consists of a mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature (usually 400° F.) becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass. Bituminous grout is composed of:

	By Weight
(*a*) Bituminous binder	45 per cent
(*b*) Mineral aggregate	55 per cent

The bituminous binder (*a*) shall conform to the following requirements:

For use above ground level, the binder shall correspond to either asphalt or coal-tar pitch (high or low carbon) suitable for use in damp-proofing and waterproofing above ground level.

For use below ground level, the binder shall correspond to either asphalt or coal-tar pitch intended for use in damp-proofing and waterproofing below ground level.

The mineral aggregate (*b*) shall be a siliceous sand, which shall conform to the following:

	By Weight
Passing 20-mesh sieve	100 per cent
Passing 200-mesh sieve.....Max.	5 per cent

A grout composed of distilled coal tar with heated sand is spread evenly in a layer 1¼ to 1½ in. over concrete floors in basements or cellars where the ground is damp, and creosoted planks of wood are embedded in the plastic mixture, before it cools by hammering into place and then toe-nailing together. The finishing layer of floorboards is then nailed upon the planks at right-angles or diagonally. This method of treatment is known as the "sub-floor tar" method.[23]

Bituminous Enamel for the Inside of Steel Ships. The bituminous enamels are used to protect the inside of steel hulls of vessels from the corrosive influence of bilge water, as well as from the natural causes of corrosion. They are also designed to resist abrasion.[24]

The bituminous enamel consists of two materials, the priming solution and the enamel proper. The priming solution is composed of a relatively high fusing-point bituminous substance, preferably of low susceptibility to temperature changes, dissolved in sufficient volatile solvent to impart a brushing consistency. The enamel proper consists of a bituminous substance similar to that used in the primer, to which is added a finely powdered mineral filler for the purpose of increasing the resistance of the enamel to abrasion and to flowing or sagging at elevated temperatures. The primer is applied cold, and is intended to promote the bond between the enamel and the metal. The enamel is brushed on while hot over the properly dried priming coat.

The following specifications have been proposed for this purpose: [25]

General. The bituminous primer and bituminous enamel shall be made of high-grade materials and shall be so manufactured as to produce a coating which: (1) will possess good adhesive qualities, (2) will not flow or sag from a vertical surface at the highest prevailing temperature (140° F.), and (3) will not become brittle at 32° F.

Bituminous Enamel. The enamel shall consist of a homogeneous mixture of bituminous material and finely divided mineral matter. The total amount of mineral matter contained in this enamel, as determined from the ash on incineration shall not exceed 40 per cent, nor be less than 15 per cent by weight. Within the above limits the satisfactory working-qualities of the enamel shall control the quantity of mineral matter to be used. The type of mineral matter shall be left to the discretion of the manufacturer, except that slaked lime or calcium carbonate shall not be used. Such mineral matter as is added must pass a sieve, the openings of which are not greater than 0.14 mm. (0.0055 in.), which corresponds to about a 100-mesh sieve.

Bituminous Primer. The primer shall consist of a like bituminous material, containing no added mineral matter, thinned with a solvent other than benzol to satisfactory brushing consistency. It shall dry at least to tacky state in 3 hours. The flash-point of the primer shall be not less than 30° C. by the Abel closed tester. (Test 17*c*.)

Covering Power. The materials shall have the following covering power: Bituminous Primer, 1 gal. shall cover not less than 200 sq. ft. of surface over metal.

Bituminous Enamel, 1 lb. shall cover not less than 2 sq. ft. of primed surface 1/16 to 1/8 in. thick.

Workability. The enamel and primer shall be applied to a clean smooth, soft steel plate, 30 cm. by 30 cm. by 1.6 mm. (12 by 12 by 1/16 in.) in the manner best adapted for its use, leaving a narrow uncoated border about the edge of the plate. The plate shall first be coated with the primer and when this has dried to a tacky state, the enamel shall be spread at not exceeding 350° F. in a coating 3 to 5 mm. (1/8 to 3/16 in.) thick. The spreading and working qualities, together with the character of the resulting surface, shall determine its workability.

Flow Test. The plate prepared for observation of the workability of the enamel shall be used for this test. Lines 1 in. apart shall be drawn horizontally across the face of the enamel and to the edge of the plate. The plate so prepared shall be suspended for five hours in a vertical position in an oven or heated chamber which is maintained at a constant temperature of 212° F. In order to secure uniformity of temperature and other conditions, the oven shall be equipped with a mechanically driven vertical shaft passed through the center of the top of the oven, from which shaft arms extend at right angles to it. On these cross-arms the coated steel plates shall be hung and the shaft revolved at 6 to 10 revolutions per minute. A thermometer shall be carried on one of the cross-arms so that the temperature of the thermometer will be the temperature to which the plates are subjected. At the end of five hours the plates shall be removed and during this test the coating must not have slipped in any part more than one-fourth inch as indicated by the lines drawn across the coating and plate.

Brittleness or Shatter Test. This test shall be made on the sample which has been used for the workability test, and shall be conducted as follows: A 750-g (1.65-lb) steel ball shall be dropped from a height of 244 cm. (8 ft.) on the coating at a point at least 10 cm. (4 in.) from the edge of the plate. The plate is to be supported in this test by a block or the floor in such a manner that there shall be support beneath the point of impact. In this test none of the coating shall be shattered from the plate nor shall radical cracks develop longer than 4 cm. (1.6 in.), from the center of the point of impact. The coated steel plate shall then be laid coating downward, on a board through which a hole about 8.9 cm. (3.5 in) in diameter has been cut. The same steel ball shall be dropped from a height of 244 cm. (8 ft.) and shall strike the steel plate over the center of the hole in the board supporting the plate. The coating must not shatter from the plate by this test nor shall cracks develop in the coating farther from the center of point of impact than the boundaries of the hole in the board.

Penetration Test. When tested by the needle penetrometer (Test 9*b*) the enamel shall show a penetration of between 5 and 15 at 77° F. and not over 20 at 115° F. Upon heating the enamel to 158° F. for five hours in an oven as provided for in the test for determining the volatile matter (Test 16), the loss in weight shall not be more than ½ per cent, and the penetration at 77° F. shall not be more than five points lower than that obtained before heating; similarly the penetration at 115° F. shall not be more than 8 points lower than that obtained before heating.

Fusing-point. The fusing-point of the enamel by the Ball-and-Ring method (Test 15*b*) shall not be less than 170° F.

Ductility. The ductility of the enamel when tested at 77° F. in the Dow mould (Test 10*a*) shall not be less than 1 cm.

The bituminous matter used in preparing the primer and enamel consists usually of an asphaltic mixture of a low susceptibility index, although a suitably prepared coal-tar pitch may also be used.

Before applying the primer, it is necessary to remove all the rust as well as mill-scale from the metal, which must be clean and free from any previous applications of paint.

The metal is first primed and when the primer has dried to a slightly tacky state it is ready for the application of the enamel. The enamel must be melted and carefully heated until it is sufficiently fluid for brushing. The temperature of the material in the kettle should not exceed 375° F. When fluid it should be mopped on quickly, as it sets and hardens very rapidly.

Bituminous Enamel for Acid-Proofing Concrete Surfaces. For lining tanks or other concrete structures to be protected against relatively strong acids for indefinite periods, similar compositions have been used consisting of a primer and enamel. These are manufactured in a similar manner, but in place of the finely powdered mineral matter, a siliceous filler must be used, which shall be resistant to hydrochloric, sulfuric and nitric acids, showing not more than 5 per cent soluble when boiled for fifteen minutes in the acids of the strengths specified below.

Resistance to Acids. The specifications for the products are the same as the foregoing, with the exception that in the workability test, the enamel and primer shall be applied to dry, clean, dust-free concrete slabs instead of to a steel plate. The test-piece made in the workability test shall remain unaffected except for a

slight dulling when exposed in different places to the action of sulfuric acid having a specific gravity of 1.3 (about 40 per cent), nitric acid having a specific gravity of 1.22 (about 35 per cent), and hydrochloric acid having a specific gravity of 1.09 (about 18 per cent) for twenty-four hours.

Covering Power. This shall be the same except that in the case of the bituminous primer, 1 gal. should cover not less than 100 sq. ft. of concrete surface.

Extreme care must be taken in the application of these materials to obtain a continuous coat free from blisters and pinholes. The concrete surface should be as dry as possible and free from oil or grease and loose particles of dust. The latter shall be removed by brushing the surface with a bristle brush. The primer should then be applied and thoroughly worked into all hollows and pores. The bond between the concrete surface and the enamel coat depends to a great extent upon the proper application of the primer. Before the application of the enamel, the primed surface should be inspected, and all brown, dull or uncoated spots should be touched up. When the primer has dried to a slightly tacky state it is ready for the enamel.

For tanks below ground or those in which the temperature never exceeds 100° F., materials with a lower melting-point may be made from a softer bituminous substance on account of its greater ease in application. For tanks in which the temperature range does not exceed 30° F. a slightly more brittle material may be used, which although shattering under the impact of a 750-g. ball from a height of 244 cm. will withstand its impact from a height of 183 cm.

Another procedure consists in protecting concrete vessels with a layer of asphalt, a layer of cork, wire-mesh, cement-mortar, and a final coating of asphalt.[26] A mixture of asphalt or coal-tar pitch, gutta-percha, linseed oil, resin and sulfur has also been recommended for acid-proofing concrete.[27] A mixture of blown petroleum asphalt, gilsonite and mineral filler, rolled into sheets, which are softened by heating and pressed against the surface to be protected.[28]

Pipe-dips and Pipe-sealing Compounds. *Pipe Dips.* To prolong the life of metal pipes, it is sometimes customary to treat them with solid bituminous compositions [29] to protect them either inside

or outside, or both. The treatment may consist in pouring or spraying the melted compound on the pipe, or by simply dipping the pipe in the melted compound,[30] or by a combination of dipping and wrapping with a bituminized fabric, or burying the entire pipe in a trough filled with bituminous matter.[31] Steel pipes are more susceptible to corrosion than cast-iron pipes, since the minute particles of graphitic carbon or else the molecular structure of the cast iron tends to retard its action. Pipes are prone to corrode internally when used for conveying water, including water-mains or water-supply pipes. The external corrosion is brought about by one or more of the following circumstances:

(1) Exposure to moist soil conditions.
(2) Electrolysis induced by stray electric currents.
(3) Exposure to mineral salts in the soil, as for example the "alkali" normally occurring in some of the western States.
(4) Contact with dilute acid, as for example sulfuric acid occurring in the water of coal and other mines, and derived from the sulfur present in the mineral (sulfides, sulfates, etc.).

Cast-iron pipes are rarely protected at the present time, but steel pipes are often coated, and especially when used for the following purposes, viz.: water mains, lines for conveying oil across the continent, steel flumes for irrigation or power purposes, compressed-air pipes for operating block signals or switches on railways, conduits for transmission and telephone wires, pipes for conveying illuminating or natural gas, pipes apt to come in contact with acid liquors in mines, etc.

To fulfil its function satisfactorily, a covering should be:[32]

(1) Impervious to air and moisture, and a non-conductor of electricity.
(2) Of such a character that should the surface be broken through, the covering will not accelerate corrosion.
(3) Not susceptible to being chipped or broken through by any treatment which the pipe may receive during its installation or use.
(4) Durable without becoming porous, brittle or cracking by the action of air, moisture or light under the conditions to which it is likely to be exposed.
(5) Incapable of producing any deterioration in the metal to which it is applied.
(6) Easy of application.

(7) Easily renewable, if the surface becomes broken or damaged by accident or otherwise.

The following methods are used:[33]

(1) Bituminous lacquers and enamels.
(2) Bituminous dispersions (sometimes containing inhibitors, e.g., chromates).
(3) Molten bituminous pipe-dips.
(4) Bituminous mastics.
(5) Bituminized fabric wrappings, including:
  (a) Rag-felt impregnated with asphalt.
  (b) Asbestos-felt impregnated with asphalt or coal-tar pitch.
(6) Combinations of (3), (4) and (5).

The first compound to be used for this purpose was composed of moderately hard coal-tar pitch combined with linseed oil.[34] The pipe to be treated was brought to a temperature of 300° F., and then immersed into a bath of the pitch mixture maintained at the same temperature. The pipe upon being removed was kept at 300° F., and protected from draughts until the coating baked hard and tough. Pipes treated in this manner are said to have proven very durable.

Modern practice is very similar to that described above, but when coal-tar pitch is used the linseed oil is replaced with creosote oil (i.e., dead or anthracene oil), of which small quantities are added from time to time, to maintain the pitch at the proper fusing-point and hardness, since continuous heating would otherwise volatilize the light oils, and gradually harden the coal-tar pitch. Under the most favorable conditions, however, coal-tar pitch and creosote oil form a brittle coating which is apt to be injured and chip or scale off in time. For these reasons asphaltic compositions are being used in place of the coal-tar pitch, and generally with better results.

The most satisfactory asphaltic compounds correspond in physical properties with the adhesive compounds used for waterproofing railroad bridges having a low susceptibility index, moderately high fusing-point, moderate hardness, great toughness, elasticity and adhesive properties. Residual oil is generally used to keep the asphalt fluxed to a uniform hardness and fusing-point.

Mixtures have been proposed consisting of coal-tar pitch with ground mica;[35] coal-tar pitch with short-fibered asbestos (e.g.,

micro-asbestos); asphalt and petrolatum, with or without a filler, surfaced with a harder layer of asphalt;[36] gilsonite fluxed with linseed oil;[37] asphalt, paraffin wax and asbestos;[38] asphalt emulsions;[39] etc.

The bituminous mixture is maintained at about 400° F., in a horizontal or vertical tank, well insulated to prevent radiation, and heated by coal, oil or producer gas. The sections of pipe are first placed in an oven through which is circulated a current of air heated to 400° F., then immersed into the bath of melted bituminous matter, allowed to drain in a heated chamber above the tank and finally cooled. The finished coating is 0.05–1.10 in. thick, and because of the "baking" to which it has been subjected, its fusing-point becomes a good deal higher than the compound originally placed in the tank.

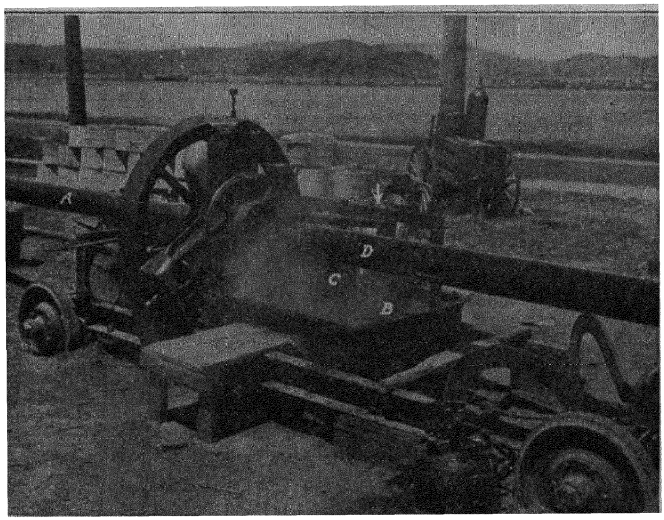
Other methods of applying the bituminous coating include: spraying the pipes with melted asphalt;[40] applying the asphalt centrifugally and consolidating under heat and pressure;[41] means for applying the asphalt to the pipes when laid in the trench;[42] applying Portland-cement mortar over the asphalt coating;[43] etc.

Pipe-Wrappings. The great objection to the use of a dipped coating is the ease with which it becomes injured on handling during transportation or installation. This objection may be overcome by wrapping the pipe spirally after it has been dipped, with strips of bituminized fabric, such as tar- or asphalt-saturated paper, felt or cloth 0.005–0.100 in. thick (measuring 10–15 sq. ft. per lb.).[44] A saturant which is claimed to resist decay and rotting consists of a mixture of soft asphalt, pine-tar pitch, creosote oil and fatty-acid pitch, with a coating composed of blown asphalt, paraffin wax and petroleum grease.[45] A machine for wrapping the pipe is illustrated in Figs. 137 and 138.[46] Another procedure consists in first dipping the pipe in asphalt heated to 290–300° F., then wrapping with plain asbestos felt, again dipping in asphalt, and finally wrapping with asphalt-saturated burlap.[47] For wrapping 2-in. pipes, 8-in. strips are used; for 4-in. pipes, 16-in. strips; for 8-in. pipes, 32-in. strips; etc. Pipes finished in this manner are much less susceptible to injury, and will last for many years even when subjected to severe conditions.

Variations consist in wrapping the pipes with bituminized asbestos-felt cemented with melted coal-tar pitch containing filler;[48]

wrapping with bituminized felt in combination with kraft paper;[49] using creped asphalt-saturated felt;[50] using cellophane with an asphaltic adhesive;[51] using bituminized fabrics with Portland-cement mortar as an adhesive;[52] using a bituminized fabric surfaced with aluminum foil;[53] etc.

Pipe-sealing Compounds. Bituminous compounds[54] have also met with considerable success for sealing the joints of metal or



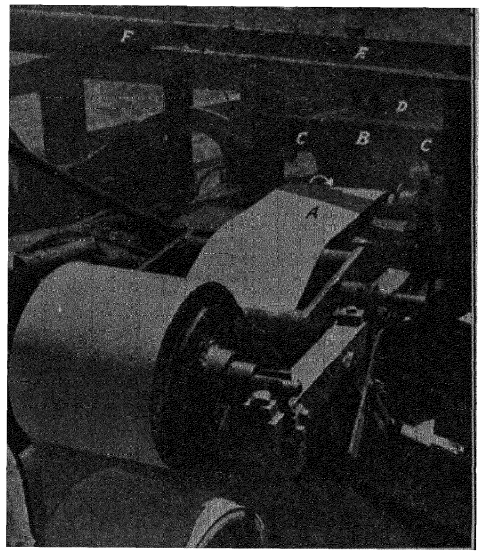
Courtesy The Paraffine Companies, Inc., San Francisco

FIG. 137.—Pipe Covering Machine in Action. *A.* Pipe before it is covered. Arrow points to uncemented felted fabric before passing to cement bath (*B*). *C.* Cemented fabric pulled upward and wound around pipe by the combined revolving and advancing movement of the pipe. *D.* Finished pipe covered with cemented felted fabric.

earthenware sewer and drain pipes. Asphaltic compounds are usually used for this purpose, carrying 50 to 65 per cent of finely divided mineral matter, preferably silica, on account of its resistance to acids and other corrosive agencies.

The pipes may be assembled underground in a trench or pipe gallery, and the bituminous composition melted at a temperature of 350 to 400° F. poured into the joints, which should first be well caulked to hold the compound in place. Sometimes two or more sections of the pipe are joined together above ground, while they are maintained in a vertical position, and when the compound cools

they are coupled to additional lengths below ground. This will save time, since it is easier to form the joints while in a vertical position, than when laid horizontally.



Courtesy The Paraffine Companies, Inc., San Francisco

FIG. 138.—Fabric-feeding Attachments of Pipe Covering Machine. *A*. Special felted fabric before treatment. Arrow points to treatment bath through which the felted fabric passes before emerging at *B* as cement-impregnated covering. Surplus cement is removed by roller *C*. Fabric passes along horizontally at *D* and then behind *E*, where it is wound around the pipe, which is shown at *F*, fully coated and protected.

The following specifications apply to a well-known pipe-seal compound on the market:

(Test 7)	Specific gravity at 77° F.	1.50 to 1.75
(Test 9*b*)	Penetration at 77° F.	40–60
(Test 9*c*)	Consistency at 77° F.	10 to 15
(Test 10*b*)	Ductility at 77° F.	Greater than 1.0
(Test 11)	Tensile strength at 77° F.	Greater than 10.0
(Test 15*a*)	Fusing-point (K. and S. method)	185–200° F.
(Test 15*b*)	Fusing-point (R. and B. method)	200–225° F.
(Test 16*a*)	Volatile matter at 500° F. in 5 hrs.	Less than 1.5 per cent

The principal considerations are that the compound should possess a high fusing-point so as not to soften in warm weather or on coming in contact with hot water flowing through the pipe; it should

be sufficiently ductile to permit the pipe settling in sections without the joints breaking open; and it should have great adherence and tensile strength to enable the line to expand or contract without tearing away the compound from the pipe.

The advantage in using bituminous compounds for sealing joints is because they will permit the pipe to settle, as it is very apt to do in a freshly filled trench, without danger of the joints opening. In addition, they prevent the roots of trees or shrubs working their way into the joints and congesting the inside of the pipe line. Other materials used for this purpose, such as cement mortar, sulfur, etc., possess these defects.

Electrical Insulating Compounds.[55] Bituminous compounds adapt themselves very well for electrical insulating purposes, on account of their high breakdown voltage, resistance to moisture, acids, alkalies and changes in temperature, also because they are in most cases capable of withstanding exposure to the weather.

A review of the patent literature reveals thousands of patents in the electrical industry, involving the use of bituminous materials and for hundreds of different purposes. The scope of this book will permit a brief survey of but the most important principles involved.

Semi-solid to solid bituminous compounds capable of melting under the action of heat are combined in many ways, often with the addition of other substances, including resins; rubber; mixtures of asphalt with rubber;[56] grahamite and rubber;[57] coal-tar pitch and rubber;[58] wood tar or wood-tar pitch and asphalt or rubber;[59] wax-tailing and rubber;[60] gilsonite and rubber;[61] montan wax or montan pitch and rubber;[62] mixtures containing chlorinated rubber;[63] stearin pitch;[64] montan pitch;[65] stearin pitch and cachew-nut oil heated with $(CH_2)_6N_4$;[66] mixtures of ozokerite, asphalt and resin;[67] ozokerite, polymerized vegetable oil and sulfur;[68] carnauba wax, asphalt and sulfur;[69] blown asphalt and vaseline;[70] mixtures containing mineral fillers; etc.

The electrical resistance of bituminous compounds varies from 200 to 1200 volts per mil. ascertained by subjecting a specimen at 77° F. between two spherical terminals, 2 cm. in diameter, to an alternating current of 60 cycles, the voltage being increased at the uniform speed of 600 volts per minute, until a breakdown occurs.

The following figures show the volume resistivity of solid bitu-

minous and other dielectrics, expressed in ohm-centimeters, in the order of decreasing values:[71]

Special paraffin	Over 500×10^{16}
Ceresin	Over 500×10^{16}
Hard rubber	100×10^{16}
Asphalt (medium hard)	50×10^{16}
Sulfur	100×10^{15}
Rosin	50×10^{15}
Chlorinated wax ("halowax")	20×10^{15}
Shellac	10×10^{15}
Glass	8×10^{15}
Yellow wax	2×10^{15}
Mica (brown African clear)	2×10^{15}
Unglazed porcelain	3×10^{14}
Tetrachlornaphthalene	50×10^{12}
Mica (India ruby stained)	50×10^{12}
Paraffined mahogany	40×10^{12}
Italian marble	100×10^{9}
White celluloid	20×10^{9}
Slate	100×10^{6}

The figures in Table LVI show the effect or moisture on the electrical properties of bituminous materials.[72] The capacitance and conductive measurements were made at 1000 cycles per second on a shielded bridge.

It is claimed that the dielectric strength of asphalt may be increased at least six-fold by combining with casein dissolved in a sulfonated oil.[73] Mixtures of asphalt, cellulose derivatives (e.g., acetyl-cellulose, nitro-cellulose, viscose, or esters of these substances) and camphor have likewise been described;[74] likewise mixtures of gilsonite, chlorinated naphthalene, ozokerite, carnauba wax or candelilla wax and cresol-formaldehyde resin;[75] other synthetic resins (e.g., alkyd resin) admixed with asphalt; mixtures of asphalt with lead linoleate or copper stearate;[76] asphalt with various fillers, including ground mica,[77] powdered quartz;[78] etc. Since pitches containing carbonaceous matter do not resist the electric current as well as those which are completely soluble, it has been proposed to remedy this by dissolving out the soluble constituents, filtering, and evaporating the solvent.

Vacuum Impregnating Compounds. These are used for insulating the field and armature windings of motors and dynamos, also magnet and transformer coils. The copper wire loops are wound with muslin, or in some cases with asbestos, and then impregnated

TABLE LVI

Material	Dielectric Constant			Effective A-C Conductivity (Unit = 10^{-12} mho. cm.)			Volume Resistivity (Unit = 10^{12} ohms cm.)			Physical Changes	
	Initial*	After Immersion†	Final‡	Initial*	After Immersion†	Final‡	Initial*	After Immersion†	Final‡	Final Water Content	Difference in Initial and Final Weights
										Per Cent	Per Cent
Candelilla wax	2.38–2 49	2.50–2.62	2.45–2 56	6.0–6.4	18	9.3–9.4	>290	>110	>630	0.13–0.20	0.00
Carnauba wax	2.66–2.83	3.84–4.19	2.82–2.83	4 9–5 3	240–300	74–75	60–120	0.3–4	270	0 72–0 90	0.00
Montan pitch	2.60–2 62	2.63–2.68	2.60–2.62	20–21	25–26	24	>290	75–160	>1200	0 09–0.11	+0.22 to+0.27
Montan wax	2.62–2.67	2.88–3.05	2.68	29–31	45–55	25	>290	>270	>610	0.62–0.67	−0.02
Japan wax	2.96–2.99	3.15–3.20	2.91	9–12	33–34	13	6–7	0.40–0.75	21	0.26–0.58	−1.02 to−1.4
Chinese wax	2 94–2 98	3 62–3.89	2.95–2 97	125–137	189–210	120–123	1–2	0.03–0.16	1–2	0.75–1.16	−0.53 to−0.81
Bayberry wax	3.22–3 27	10.4	3 31	89–105	6000	110	2–5	0.00054	0 96	6.1	−0.50
Beeswax	2.87–2.88	3 11–3.26	2.84–2.90	45–48	70–85	56–58	5–12	0.8–1.4	7–8	0.42–0.43	−0.13 to−0.20
Ozokerite	2.37–2 43	2.50–2 55	2.40–2.47	10–12	19–24	15	30–40	7–9	30–40	0.85–1.20	−1.0 to−1.3
Ceresin	2 16–2 24	2.29–2.32	2.28–2.29	<0.37	10–12	1.1	>290	400–>600	>900	0 04–0.05	0 00 to−0.05
Paraffin	2.21–2 24	2.31–2.38	2 24–2.27	<0.37	3–7	0 74–0.80	>290	150	>900	0.04–0.06	−0 43 to−0.66
Paraffin	2.19–2.24	2.37–2.55	2.26–2.30	<0 37	16–22	<0 74–0.94	>290	18	>660	0 06–0.11	−0 66 to−0.71
"Superla wax"	2.32–2.35	2.36–2.37	2 34–2.35	4.2–4.6	2.1	0 37	>460	>560	>1000	0 00–0.03	−0.02
"Naudella wax"	2.50	3.37	2.63	16	110	16	>610	2.6	>1000	0.50	+0.15
Zinsser's insulating wax	3.98–4 27	21	5.2 –6.5	48–60	600–2500	250–375	>490	0.00031	60–90	1 72–2.21	−0.03 to 0.00
Zinsser's insulating wax-beeswax	3.21–3.40	14.0–14.2	3.64–3.83	80–95	2000–2800	89–120	140–>290	<0.00031	130–150	1.5–2.4	−1.4 to+2.4
Paraffin-beeswax	3 50–3 55	3.55–3.61	2.53	22–26	30–39	15–18	20–27	8–10	31–33	0 11–0.54	−0.18 to−0.31
Paraffin-rosin-rosin oil-carnauba	2.45–2.47	2.58	2.48	23–24	30–32	18	>450	100–>500	>500	0.32–0.33	−0.42 to−0.48
Paraffin-rosin-hydrated lime	2.67	2.64–2.69	2.60–2.68	3.2–3.9	18–20	8	>910	>740	>900	0.17	+0.50
Zinc stearate	2 58	5 83	.....	5.7	210	.....	640	0.0003	.....	3.0	+0.45
Aluminum stearate	2.62–2 78	4.93–5 27	2.57	13–17	74–130	12	400–>900	4–12	>1000	2.7	+0.20
Blown gilsonite and residual petroleum oil	2.53–2 54	2 92–3.03	2.58	14	126	22	>290	50–95	>560	3.3	+0.63
Coumarone resin	2 79–2 80	2.87–2.92	2.96	9.0–9.7	15–17	1.0	>300	640	>1000	0.41	−0.04 to 0 00
Rosin	2 69–2.76	2.81–2.87	.....	2.4–6.2	7.4–7.7	4 2–5.0	550	300–700	... .	0 46	−0.28
Manila copal	3.05–3 09	3.22–3.38	2.63	7–15	21–23	17	>410	720	>800	0.41–0.60	0.00
Stearin pitch	3.34–3.41	3.55–3.60	3.26	67–75	77–81	57	14–18	6.5–7.0	39	1.23–1 40	+0.30 to+0.48
Soft blown petroleum asphalt	3.07–3.12	2.90–3.03	2.96	35–43	32–42	30	50–72	40–52	1000	0.47–0.52	−0.02 to 0.00
Soft blown petroleum asphalt-rosin-rosin oil	3.07–3.22	2.94–3.04	2.88	36–43	56–58	33–35	14–18	7.9	26–33	0.44–0.56	−0.05 to 0.00
Spermaceti wax	3.60–3.75	6–17	3.74–3.89	6.0–6.6	500–100,000	10–12	3–4	0.00059–0.00031	9–15	1.27–2.11	−0.37
Shellac	3.62–3.71	7.7 –>20	3.58	16–17	40,000–910,000	17	>560	<0.00031	>1000	4.3 –5.1	−1.9 to 0.00
Halowax	3.63–3.83	4.24–6.37	3.97–4.23	4.4–7.4	24–40	3–4	4–9	0.4	0 1–0.7	0.20–0.28	−0.15
Stearic acid	2.37–2.41	>20	2.33–2.38	4.4–12	>130,000	5–6	3–7	<0.00031	1–6	3.2 –4.0	−0.2 to−0.4

* After drying over calcium chloride. † After 6 months' immersion in 3.5 per cent sodium chloride solution. ‡ After redrying.

with a melted bituminous or oleo-resinous composition. The former only falls within the scope of this treatise, and includes asphaltic products composed substantially of the same materials, and having approximately the same physical properties as the surface coatings of prepared roofings. The following characteristics are of importance:[79]

(1) The mixture should melt to a liquid having the lowest possible viscosity (Test 8*a*) at the temperature at which it is maintained while impregnating the coils. Since the purpose of the compound is to penetrate the muslin wrapping of the wire, it follows that the more liquid the melted compound, the more thoroughly it will fulfil this function.

(2) The softer the compound for the prescribed fusing-point, the less it is apt to crack in service, especially as the revolving armature of a motor or dynamo is subjected to the most *extreme* conditions in regard to vibration. Should the compound crack or powder, its insulative value will be nullified, and the machine will become short-circuited and put out of commission. The mixture should preferably have a penetration of 75 to 125 at 77° F. (Test 9*b*). Its susceptibility index should also be as low as possible, and under no circumstances in excess of 25 (Test 9*b*).

(3) The ductility at 77° F. should be as high as possible, and preferably greater than 5 (Test 10*a*).

(4) The fusing-point by the K. and S. method (Test 15*a*) should exceed 180° F. This will insure the compound remaining in place when the machine heats up in service.

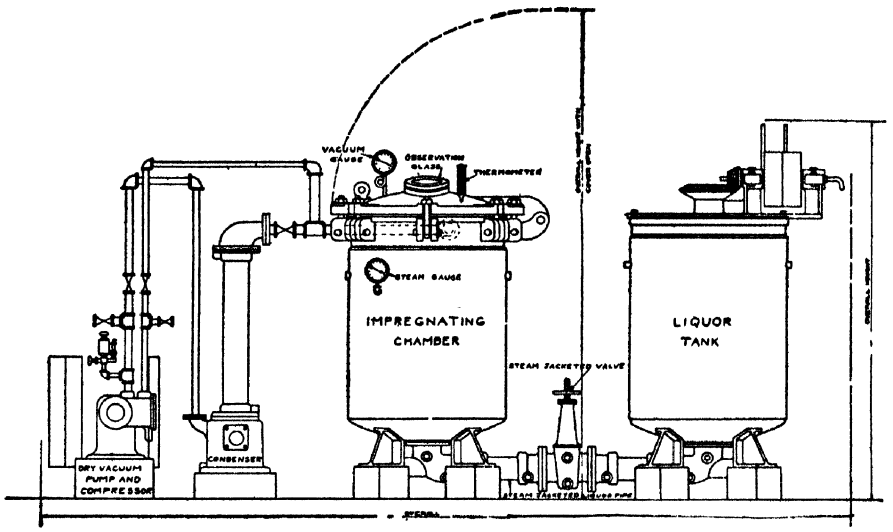
(5) The volatile matter should not exceed 2 per cent at 500° F. in five hours (Test 16).

(6) The solubility in carbon disulfide (Test 21) should exceed 99 per cent. Mineral or carbonaceous matters will interfere with the penetrating properties of the compound.

Residual oil is ordinarily used to flux the compound to its original consistency, if the hardness or fusing-point increase while maintained in the liquid condition. Coal-tar pitch may also be used, although the free carbon present is apt to inhibit the impregnation, although it is claimed that this obstacle may be overcome by first passing the melted coal-tar pitch through a colloid mill.[80] Other mixtures recommended for this purpose include: asphalt fluxed with resin;[81] manjak, esterfied rosin and china-wood oil;[82] etc.

The apparatus consists of two steam-heated, air-tight iron tanks,

one for carrying the coils to be treated, and the other for storing the melted compound, as illustrated in Fig. 139. The coils are placed in the impregnating chamber, the heat turned on and the air exhausted by the pump within ½–1 in. of the barometer, which draws out all the moisture. In the meantime the compound is melted in the steam-heated liquor tank at 300° F., whereupon it is allowed to enter the vacuum chamber and subjected to a pressure of 90 lb. per sq. in. which forces it throughout all parts of the coil. It



Courtesy of J. R. Devine Co.

FIG. 139.—Vacuum Impregnating Apparatus.

is maintained under compression for one or more hours, when the valve between the tanks is opened, the compound forced back into the liquor tank, and the excess allowed to drain from the coils upon being subjected to a dry heat for half an hour, which also completes the saturation. It is claimed that coils treated by this process give better service than those insulated by varnish, and moreover, since they become practically solid, there is no danger of the wires slipping. The total cost of treatment is less than when varnishes are used, but to offset this, the equipment is more expensive. The units are made with impregnating and compound storage chambers measuring 108 in. diameter by 240 in. high as a maximum.

Transformer and magnet coils may be treated in the same manner, and tests made in the author's laboratory demonstrate that

tightly wound coils several inches thick, composed of high-gauge wire, may be thoroughly impregnated. Bituminous compounds should be used for air-cooled transformer coils only, as they will become softened and dissolved in oil-immersion transformers. The method is now being used almost universally in large plants.

The following products may similarly be impregnated with bituminous substances under vacuum, followed by pressure: wooden pipes for conveying liquids, wooden storage-battery boxes, cotton belting, compressed wood-pulp in various forms, fibrous conduits and tubes,[83] other bibulous bodies;[84] porous building blocks and baked clay articles of various shapes;[85] asbestos-cement panels (known as Ebonized lumber) for electrical insulating purposes;[86] stone or concrete may be impregnated with bituminous dispersions;[87] etc. A mixture of asphalt and paraffin wax has been suggested for the foregoing purposes.[88]

Cable-splicing and Pot-head Compounds. These are used for filling the metal receptacles in underground electrical transmission lines, where feeders branch off from the main wires. The connections are usually made inside of a metal box known as a "junction box" or "pot-head." Asphaltic compounds fusing in the neighborhood of 200° F. (K. and S. method, Test 15*a*) are melted and poured into the receptacle to hermetically seal the wires, after the junction or connection has been effected.

Battery-box Compounds. These are used for sealing "dry" batteries.[89] After the zinc container is filled with chemicals in paste form, and the rod of carbon introduced, the top is hermetically sealed with the "battery-box compound." This consists of moderately hard coal-tar pitch (fusing at 160 to 170° F. by the cube method, Test 15*c*) combined with about an equal weight of siliceous filler.

"Carbons" for Batteries, Electric Lights and Armature Brushes. "Carbons" for the electrical industry are formed by heating a mixture of powdered coke and hard coal-tar pitch (fusing above 400°F. by the cube method, Test 15*c*), in a closed metal mold. Upon subjecting this mixture to a red heat, the coal-tar pitch carbonizes and consolidates the particles of coke-carbon. Hard coal-tar pitch suitable for this purpose is obtained by distillation in a still through which a current of inert gas, such as nitrogen, is circulated. This

produces a pitch fusing above 400° F. and which will not foam upon being coked at 700° F. or higher.[90] Electrodes for use in metallurgical industries [91] are manufactured from gas-retort carbon, anthracite, tar coke or petroleum coke. The form of carbon selected is first broken into lumps and roasted in a kiln at 2000° C., to remove the last traces of volatile matter; then washed to remove the water-soluble salts; dried; ground to produce particles between 8- and 100-mesh; and mixed with a small amount of the coal-tar pitch in a steam-jacketed mixing vat. This mixture is then passed through dies of the requisite diameter, by a steam-heated hydraulic press, and the rods are calcined in a gas-fired air-tight kiln at 1500 to 1600° C. for six to sixteen days, depending upon the size of the electrode, until the whole mass "slags." The electrode is allowed to cool and brushed to a smooth surface. Another method consists in heating the coal-tar pitch in closed molds at 600° F. under several atmospheres' pressure and then suddenly reducing the pressure to atmospheric, without further application of heat, whereupon vapors are evolved and a residue of coke remains.[92] Clay pigeons are produced in a similar manner. Armature carbons are often mixed with a proportion of graphite to reduce their friction against the rapidly revolving armature; and electric light carbons with a proportion of mineral constituents (usually less than 20 per cent) including the rare earth oxides, silicates, fluorides, borates, bauxite, etc., to increase the luminosity or modify the color of the electric arc.

Bituminous Rubber Substitutes. On account of the high price of pure rubber, it is often adulterated with fusible materials derived from the animal, vegetable or mineral kingdoms. Resins, animal or vegetable oils and fats, animal and vegetable waxes have been used largely for this purpose. Many tons of bituminous compounds, known to the trade as "mineral rubber," abbreviated "MR," have found a ready market. The following classes of bituminous substances have been used for this purpose:

(1) Ozokerite and paraffin wax.
(2) Hard native asphalts.[93]
(3) Asphaltites either used alone or fluxed.[94]
(4) Blown petroleum asphalt.
(5) Wurtzilite asphalt.

(6) Rosin pitch.
(7) Fatty-acid pitches.[95]
(8) Special products including chlorinated naphthalene, etc.[96]

The bituminous materials are regarded mainly as "extenders," diluents or cheapeners, although in some cases they are purposely used to soften the rubber, and in others to increase its weather-resisting properties. Rubber substitutes, marketed under the name "factis" or "faktis" are prepared by vulcanizing a mixture of asphalt with vegetable oil or fatty-acid pitch, with sulfur or sulfur dichloride (in the cold).[97] At the present time, mineral rubber consists of blown asphalts derived from semi-asphaltic or asphaltic petroleum, either as such, or fluxed with gilsonite either before or after the blowing process. Experiments made with mixtures of rubber with blown mid-continental residual oil fluxed with gilsonite gave the following results: a beneficial effect was noted upon the physical properties in proportions ranging between 3 and 15 volumes of M.R. per 100 volumes of rubber. Beyond 15 volumes, a decided slowing up in the "recovery" was observed, although the eventual "recovery" was the same as for pure rubber.[98]

These substitutes are incorporated with the raw rubber on the masticating rollers (consisting of heavy steel rollers revolving at unequal speeds, capable of being heated by steam or cooled with water). One method consists in first combining the substitute with reclaimed rubber and mineral fillers on the rolls, the raw rubber is then masticated a short time, whereupon the bituminous mixture, still warm from the masticating process, is gradually worked in, small quantities at a time, and the composition finally vulcanized in the usual manner.

The most satisfactory bituminous materials for this purpose include:

(1) Products which of themselves possess "rubber-like" properties (including a certain degree of toughness, resilience, tenacity and ductility), as for example gilsonite combinations, wurtzilite asphalt.[99] blown petroleum asphalts, and certain of the fatty-acid pitches,[100] and in addition will not adversely affect the resilience and tenacity of the rubber itself.

(2) Bituminous substances which are capable of being hardened and toughened by the sulfur used in the vulcanization process. To this class belong the gilsonite combinations, wurtzilite asphalt and fatty-acid pitches.[101] High boiling-point resinous distillates (fusing at 110–190° F. by the cube method, and having a specific gravity of 1.15 to 1.25 at 60° F.) obtained by destructively distilling coal-tar pitch above 700° F. have also been proposed.[102]

(3) Bituminous products that will flux or blend completely with the rubber, and will not separate or "sweat out" of the mixture on aging. Sometimes fluxed with lanolin, vaseline, wood tar, stearic acid, etc.

The harder waxes and bituminous materials [103] are used in mixtures of hard rubber (ebonite), and the softer waxes and bituminous materials in soft rubber compositions.[104] Sometimes the bituminous product is partially vulcanized with sulfur before it is combined with the rubber, especially when too soft to be used without further treatment.

The following types of mixtures have been proposed, viz.: asphalt, rubber, linseed oil and sulfur; [105] asphalt, rubber, sulfur and mineral filler; [106] asphalt, rubber, linseed oil and zinc oxide; [107] asphalt heated with a rubber fabric until the rubber dissolves; [108] asphalt, coal-tar pitch and rubber; [109] coal-tar pitch, rubber and sulfur; [110] coal tar, rubber and fillers; [111] water-gas tar, coke-oven tar, rubber and sulfur; [112] coal-tar pitch, rubber and a colored mineral pigment; [113] asphalt, rubber, magnesium oxide, sulfur and antimony sulfide; [114] rubber combined with "asphaltenes" derived from asphalt; [115] paraffin wax and rubber; [116] blown asphalt, rubber and gum thus; [117] asphalt, rubber, rosin and linseed oil; [118] asphalt, rubber, ozokerite and sulfur; [119] asphalt, rubber and fibrous filler vulcanized with S_2Cl_2; [120] asphalt, rubber, coal tar, china-wood oil and glycerin; [121] fatty-acid pitch, rubber and linseed oil; [122] fatty-acid pitch, ozokerite or ceresin heated with picric acid; [123] fatty-acid pitch, rubber, gelatine, fillers and sulfur; [124] blown petroleum asphalt, pine-tar pitch, rosin, gum arabic (gum tragacanth or glue) and pigments; [125] blown petroleum asphalt and desiccated sulfite liquor; [126] asphalt, casein and fillers; [127] asphalt, oleic acid, gelatine, glycerin, tannin and soda; [128] wood-tar pitch, rubber, shellac,

wax and fillers;[129] a blown mixture of residual oil, rubber and pine tar;[130] etc.

Molded Compositions.[131] *Mixtures for Small Molded Articles.* Formerly shellac was extensively employed for manufacturing small molded articles used for electrical fittings, push-buttons, knobs, handles, filling teeth;[132] dust-guards;[133] etc., but this is being substituted by asphalts and asphaltites.

Such mixtures are composed of four classes of materials, viz.:

(1) Hard native asphalts, asphaltites, or wurtzilite asphalt; also hard coal-tar pitch, various waxes[134] (e.g., ozokerite, montan wax, or higher melting vegetable waxes), etc.

(2) Vulcanized rubber or resinous substances, including rosin (used alone or hardened by heating with lime, oxide of zinc, litharge, etc.), shellac (sometimes added in a small proportion),[135] Manila copal, Congo copal, Kauri copal, damar, synthetic resins, etc.

(3) Sometimes vegetable drying oils[136] are added to toughen the mixture. Oleic acid[137] is claimed to prevent adhesion to the dies. A mixture of fatty-acid pitch, aluminium palmitate and wax has likewise been proposed.[138]

(4) Fillers, including the products referred to in Chapter XXIV. Clay is claimed[139] to give superior results.

The following represent a few of the mixtures that have been proposed: gilsonite, powdered wurtzilite, blown petroleum asphalt and filler;[140] gilsonite and an alkyd resin;[141] coal-tar pitch, a synthetic resin and asbestos;[142] asphalt, china-wood oil, asbestos and a solvent (e.g., petroleum naphtha and furfurol);[143] mineral pigments may be added to impart color to the mixture;[144] water may be added to improve the gloss of the finished product;[145] asphalts mixed with 15 to 40 per cent by weight of sulfur;[146] asphalt emulsions;[147] etc.

Innumerable combinations are used for this purpose, all of which are based upon the following general principles:

As the molded article is usually subjected to severe usage, it is fundamentally important that the ingredients should possess great tensile strength and toughness. They should be sufficiently hard to

withstand deformation under pressure, and fuse sufficiently high not to soften in the sun or any heat to which they may be subjected in use.

The asphalts and asphaltites used should accordingly have a fusing-point not lower than 190° F. (Test 15*a*), a penetration at 77° F. (Test 9*b*) of not exceeding 5, and a tensile strength at 77° F. of not less than 10 (Test 11). The function of the resinous matter is to enable the bituminous constituents to melt up freely and impart gloss to the finished product. The powdered mineral matter hardens and toughens the mass, increasing its tensile strength and making it less susceptible to deformation under pressure. The fibrous matter binds the mass together and enables it to stand sharp blows without fracturing.

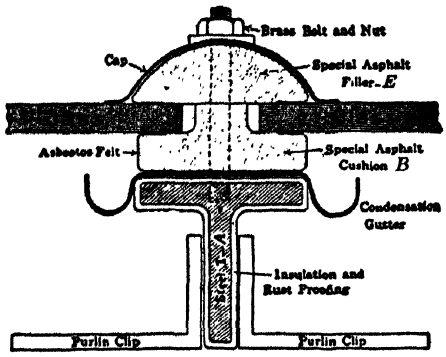
The constituents are combined by melting together the bituminous and resinous bodies, and then stirring in the powdered mineral matter with or without the fibers. When the mixture is uniform it is allowed to cool and then granulated.[148] The granulated mixture is then measured or weighed into the mold and subjected while cold to a pressure of 2000 to 5000 lb. per sq. in., either when cold,[149] or while still hot and then allowed to cool rapidly.[150] The article is then removed from the mold and baked in an oven to raise the fusing-point of the binder by the expulsion of a portion of the volatile constituents, accompanied by polymerization or oxidation. The baking process is discontinued at a point when the composition no longer softens.[151]

Alternate procedures consist in extruding through a die while hot and plastic;[152] mixing with a mineral filler, powdering and molding, either hot or cold;[153] compressing under a pneumatic hammer while cold;[154] calendering between sets of rollers while cold;[155] etc.

Most compositions of this character are naturally dark in color, due to the bituminous matter present and they are often darkened still further by adding a small proportion of black pigment, such as lamp-black or carbon-black. In other cases, the mass may be colored deep shades of red, brown or green by incorporating an intense pigment of the corresponding color.[156] Mixtures of this character have been proposed for use as sealing wax. Brightly colored mixtures are not obtainable, except by forming a laminated structure composed of a bituminous base molded to a light-colored, pigmented nitrocellulose facing.[157]

Preformed Joints and Washers. A bituminous product has recently met with considerable success in glazing and sky-light con-

struction,[158] consisting of an asphaltic cushion enveloped in sheets of asbestos, in which the panes of glass are mounted and securely bolted together, as shown in Fig. 140.



Courtesy of Asbestos Protected Metal Co.

FIG. 140.—Preformed Washers for Skylights.

A represents a steel T-beam, protected with galvanizing or other rust-proofing. The condensation-gutter is composed of sheet steel joined to a waterproof asbestos envelope by an asphaltic composition. The asphaltic cushion and cap-filler *E* are encased in waterproofed asbestos, and forced together in between the two sheets of glass, forming a pliable, resilient, water-tight but firm joint, which prevents the glass from being subjected to injurious strains. The upper cushion is reinforced with a cap of asbestos-protected steel, and fastened with a bolt and nut.

The cushions *B* and *E* are composed of a fluxed asphaltite or a blown petroleum asphalt of high fusing-point (in the neighborhood of 250° F.), low susceptibility index (under 20) and absence of brittleness (hardness at 77° F. less than 20—Test 9*c*), mixed with 10–15 per cent of fibrous asbestos, which binds it together in a tough and non-breakable mass. These strips are further protected by being joined to an enveloping sheet of asbestos felt, waterproofed with a vegetable drying oil or bituminous saturant, similar to that used in manufacturing prepared roofing.

Molded Brake-linings, Clutch-facings and Friction Elements. These are composed of asbestos fibers and fillers (e.g., graphite, talc, powdered lead, etc.) and asphalt or fatty-acid pitch, synthetic resins, vegetable drying oils, etc.[159]

Bituminated Cork Mixtures. Bituminated cork blocks or tiles have been produced of asphalt, granulated cork and mineral filler [160] including granulated charcoal.[161] In Europe, sheets have been prepared for insulating refrigerator plants, ice-chests, cold brine and ammonia pipes,[162] composed of 45 to 50 parts by weight of finely powdered cork and 100 parts of bituminous binder. The composi-

tion is prepared in a steam-heated mixing machine. The binder may consist of asphalt, coal-tar pitch or an aqueous emulsion of soft coal-tar pitch with colloidal clay.[163] An alternate procedure consists in moistening the particles with water and then adding the bituminous binder dissolved in a volatile solvent.[164] The mixture is pressed into various forms, such as plates, slabs or hemi-cylindrical sections suitable for covering pipe. It has a specific gravity between 0.25 and 0.40 and forms a good insulator against heat and cold.

Bituminated cork compositions are also used extensively in the United States under the name of "shoe fillers" for filling the insoles of shoes, and composed of a mixture of coarsely ground cork, with wax tailings, residual asphalts, or an emulsion of asphalt with glutinous substances.[165]

The mixture is trowelled between the inner and outer soles of shoes while hot and plastic, and allowed to set by cooling. It serves to insulate the bottom of the foot, and incidentally to replace the more expensive leather.

The use of bituminized felt, cut to the proper shape;[166] also a mixture of ground roofing waste and rubber;[167] likewise fibrated asphalt compositions,[168] have been proposed for making shoe fillers and insoles.

Battery Boxes.[169] In recent years these have been developed to a high state of perfection, and are now used successfully in automobiles and for other purposes where a small sized container is required. One composition is prepared by first mixing asphalt and infusorial earth in one mixer, and asphalt and fibers (e.g., cotton linters) in another; then mixing the two together while hot, and utilizing the mixture for molding purposes.[170] Another consists of a mixture of amphibole asbestos (25 per cent), silex or silocel (25 per cent), gilsonite (25 per cent), Mexican asphalt fusing at 200–210° F. (20 per cent) and montan wax (5 per cent).[171]

Another composition consists of gilsonite (with or without the addition of hard coal-tar pitch) 25 per cent by weight, hard residual asphalt 18 per cent by weight, silocel (i.e., calcined infusorial earth) 43 per cent by weight, and cotton linters 14 per cent by weight. The gilsonite and hard residual asphalt are first fluxed together (R. and B. fusing-point 225–235° F., penetration at 77° F. 0) and then

when the mixture has cooled to a thick and stringy state, the cotton linters and silocel are incorporated in a Werner-and-Pfleiderer type of mixer. The addition of a few bucketsful of water during the mixing process serves to stiffen the mass and result in a more thorough and uniform distribution of the fibers, thereby increasing the strength of the finished product about 10 per cent.

Fibrated Bituminous Compositions.[172] These are prepared by combining comminuted fibers with a bituminous binder by grinding the fibers or otherwise mixing them with the binder (which may be either hot or cold), with or without the use of water, in the same manner as described in the following chapter on Bituminized Fabrics, under the section entitled "Saturating the Fabric." These mixtures may likewise be prepared from the waste which results in the manufacture of prepared roofings and composition shingles by warming the shredded waste and extruding same through a perforated drum;[173] chilling the material and then grinding the mass;[174] boiling the waste with 5 per cent of alkali until disintegrated;[175] boiling with clay and water under 80 lb. steam pressure;[176] etc. Other mixtures include the following, viz.; fibers and water masticated with bituminous substances and molded under heat;[177] ground gilsonite and asbestos fibers, which when ready for use is mixed with a flux that will combine with the gilsonite and thereby cause the mass to set;[178] asphalt emulsions mixed with comminuted fibrous material;[179] asphalt emulsion with mineral wool (e.g., rock wool or slag wool);[180] powdered asphalt incorporated with rock wool by means of a steam-jet;[181] blowing the fibrous constituents into a flowing stream of the melted asphalt;[182] impregnating the bibulous material with the melted asphalt under vacuum, followed by pressure;[183] etc.

The mixture may be molded, after drying, as previously described; or it may be extruded through a die under pressure while in a heated, plastic state.[184] Among other uses, fibrated compositions have been recommended for use in tree-surgery,[185] for packing pipe-joints;[186] surfacing miniature golf courses,[187] etc.

Roof Tiles. Attempts have been made, but so far without commercial success, to produce roof tiles from molded mixtures of gilsonite and other asphalts with fibrous vegetable or mineral matter;[188] Kentucky rock asphalt and Portland cement,[189] or by heat-

ing to incandescence a mixture of bituminous matter with coal dust, or ashes, with or without asbestos fibers; [190] asphalt, exfoliated vermiculite (zonolite) and clay; [191] asphalt, hemp linters, ground tanbark, sand and sulfur; [192] coal-tar pitch and clay baked until hard; [193] etc. Mixtures of this general character are first prepared in a suitable masticator, either hot or cold, and then molded under pressure, either through squeeze rolls, [194] or otherwise.

The product may be reinforced with a backing of bituminized felt; [195] or with metal; [196] or with wooden laths; [197] or the surface may be coated with asphalt in which slate granules or colored sand are subsequently embedded; [198] or the surface may be glazed with fusible glass; [199] or covered with a layer of sheet metal; [200] etc. Molded units have also been proposed for roof copings, [201] as well as in the form of an interlocking roof tile. [202]

Floor Tiles. [203] The use of molded compositions has similarly been described for manufacturing floor tiles, including fluxed gilsonite or Sicilian rock asphalt (which contains a light-colored impregnation) and mineral pigment; fatty-acid pitch (with or without asphalt), wood flour and a colored pigment; [204] fatty-acid pitch with gilsonite or cumar resin, also asbestos and a colored pigment; [205] asphalt and mineral filler; [206] coal-tar pitch and mineral filler; [207] asphalt emulsion and powdered limestone filler; [208] etc.

The mixture is prepared in a Banbury type of mixer at 400° F., and while hot is rolled out between "sheeting rolls" and then cut into slabs, which in turn are calendered to the desired thickness. The sheets are then cut into tiles by means of block dies and cooled. The excess material cut from the sheets is returned to the sheeting rolls and used over again. [209] A machine has been patented for molding floor tiles under pressure. [210]

The following specifications have been standardized: [211]

Size. Shall be square, rectangular, hexagonal or octagonal, in dimensions as specified, from 3 by 3 in. to 12 by 36 in.

Thickness. Shall be either ⅛ in. or 3/16 in. at 77° F. ± 0.005 in.

Texture. Shall have a smooth, calendered wearing surface, free from grit and lumps.

Color. Shall be colored throughout, either in solid colors, or mottled, as specified.

Indentation. When subjected to a pressure of 30 lb. on

a ball or hemispherically ended rod having a diameter of 0.25 in., the tile shall show the following indentations:

(a) At 77° F.: At the end of 1 minute 0.009 to 0.20 in. At the end of 10 minutes 0.0124 to 0.0265 in.

(b) At 115° F.: At the end of 30 seconds not more than 0.038 in.

Impact. Shall not break or crack at 77° F. (± 2° F.) when a specimen 6 by 6 in. is supported on three 1-in. diameter steel balls equally spaced in a circle 5 in. in diameter on a steel base, and subjected to the following test:

(a) For 5/16-in. tile: A steel cylinder 1 in. in diameter, the lower end of which is hemispherical, weighing 0.350 lb. (± 0.005 lb.) shall be dropped from a height of 4½ in.

(b) For ⅛-in. tile: A spherical steel ball 1 in. in diameter, weighing 0.143 lb. (± 0.002 lb.) shall be dropped from a height of 4½ in.

Flexure. Pieces of the tile 2 in. wide shall at 77° F. (± 2° F.) sustain without breaking, both across and with the grain, a load applied at the center of an 8-in. span, of 6½ lb. for 3/16-in. tiles and 3 lb. for ⅛-in. tiles, and at fracturing, the deflection shall not be less than 0.4 in. in either case. The loading shall be so regulated that the deflection takes place at a uniform rate of 4 in. per minute.

Curling. A 6 by 6-in. specimen shall not curl more than 0.05 in. when subjected at 70° F. to 120 hours' exposure to moisture, upon being supported on a flat slab of limestone or other similar material (having a water absorption of 4 to 8 per cent by weight) partially immersed in water, with its water surface ¾ to 1 in. above the water level.

Flammability. The tile shall not continue flaming for more than 30 seconds upon being subjected for 15 seconds to a yellow gas flame 1½ in. high emanating from a ⅜-in. Bunsen burner, above which the tile is supported vertically with its lower edge ¾ in. above the top of the burner.

Modifications in the composition of the tiles include the following: Embedding grit on the underside of the tiles to facilitate adhesion when laid in cement-mortar;[212] mounting the molded tiles on a sheet of asphalt-saturated felt;[213] mounting the molded tiles on a Portland-cement backing;[214] mounting small squares of stone on a backing composed of molded fibrated-asphalt;[215] etc. Variations consist in mounting small units of asbestos-cement composition,[216] or small units of ceramic tile[217] on a backing composed of

asphalt-saturated felt. Molded tiles have also been recommended for lining concrete conduits.[218]

Floor and Step Treads. Mixtures of asphalt with grains of corundum, emery, silicon carbide, fused alumina, iron filings, or the like, are molded and affixed to a foundation of concrete, so as to prevent the pedestrians from slipping.[219]

Artificial Lumber and Railroad Ties. A molded composition has been described for this purpose, consisting of asphalt (8–11 per cent), stone dust (16 to 25 per cent), fine gravel (54 to 71 per cent) and powdered metallic iron (5 to 10 per cent), compressed at 325° F. under a pressure of 1500 lb. per sq. in., and in certain cases reinforced with metallic wires,[220] or with a core of concrete. Other variations consist of a molded mixture of asphalt and fibrous matter, with or without the addition of mineral filler;[221] concrete impregnated with molten asphalt under vacuum and pressure;[222] a core of concrete faced with an asphaltic mixture;[223] a core of concrete coated successively with layers of asphalt emulsion and melted asphalt;[224] etc.

Pipes and Conduits. These may be molded of compositions of which the following may be cited as examples: asphalt or coal-tar pitch with mineral fillers;[225] asphalt, sand and sulfur;[226] asphalt with animal or vegetable fibers;[227] emulsified asphalt with graded mineral aggregate;[228] etc. It has also been suggested that glass pipes be coated with asphalt and wrapped with paper to protect them from injury in chemical factories.[229]

Burial Vaults. These may be constructed by molding a mixture consisting of asphalt- or coal-tar-pitch mastic containing a mineral aggregate;[230] or a similar mixture reinforced with wire mesh;[231] or concrete waterproofed on the surface with a layer of asphalt mastic.[232]

Phonograph Records. It has been suggested that these be made of gilsonite fluxed with residual asphalt, mixed with asbestos and infusorial earth, and molded under pressure;[233] or an asphaltic composition as base, faced with cellulose ethers.[234]

Bituminated Leather Mixtures. Shredded leather waste and asphalt have also been combined in various ways[235] and proposed for various purposes, as for example covering floors, but these have only found a limited use owing to the high price of leather.

Briquette Binders.[236] Coal-tar pitch is most generally used for briquetting coal-dust or coke-breeze. The coal or coke powder is mixed with approximately 6–8 per cent of coal-tar pitch. Two methods are in vogue, one in which the powder is mixed with a moderately soft pitch, added in the melted state, and the other, in which it is mixed cold with pulverized hard pitch. In either case, the briquettes are formed at high compression under the influence of heat.[237] The soft coal-tar pitches have a fusing-point between 140 and 170° F. (cube method) and the hard pitches between 200 and 225° F. (cube method).[238]

Softer grades of petroleum asphalt may be used for this purpose if first mixed with 2½ to 5 per cent of powdered bicarbonate of soda, whereby on heating, carbon dioxide is liberated, converting the asphalt into a foam, which is then suddenly cooled. The mass may then be pulverized and used as a binder in the same manner as coal-tar pitch.[239] Other materials suggested for use as binder include gilsonite [240] and a mixture of asphalt with sulfur.[241]

Core Compounds. Coal-tar pitch is used similarly as a binder for the sand cores in forming iron and steel castings. A powdered hard pitch, fusing at 225–260° F. (cube method) is granulated [242] and then mixed with the sand and finally compressed under the influence of heat, to form the core.

Miscellaneous Bituminous Products. *Bituminous Fuels.* The non-asphaltic pyrobitumens constitute the principal source of solid fuels for combustion on a grate. Liquid bituminous fuels are also used as "fuel oil," including crude petroleums, petroleum residues, by-product tars, and distillates of little commercial value produced in refining petroleum and tar. These are atomized under the boiler with a steam jet.

So-called "colloidal fuel" consists of about 68 per cent of fuel oil, 30 per cent of powdered coal (e. g., anthracite, bituminous, lignite, coke, culm, screenings, etc.), and 2 per cent of a "fixateur" or dispersing agent, designed to hold the particles in suspension (e. g., lime-rosin soap, or coal-tar creosote, or a mixture of the two). The colloidal fuel contains about 1½ per cent of water, and upon storing at 40 to 55° F. requires no mechanical agitation until after about three months.[243]

A detailed description of the technology of fuels does not, however, fall within the scope of this treatise.

Tars and Oils for the Flotation of Ores.[244] During the last decade the flotation process has become an important factor in the concentration of certain minerals, including sulfides of copper, lead, zinc, silver, iron and other metals, also pure gold, gold telluride, native silver and native copper. The best results are obtained with sulfide ores. Oxide ores are not amenable to it unless they are first converted into the sulfide superficially, by a preliminary treatment with sodium sulfide.

In carrying out the process, the ore is reduced to a fine powder, 50-mesh material being usually the limit of coarseness. This is then beaten into a froth or foam with water and a so-called "flotation oil," which causes the heavier ore-mineral to rise to the surface, forming a pulp with the froth, and the lighter gangue-mineral to settle out. The flotation process has accordingly been aptly termed "concentration upside down." The "pulp" when it becomes thoroughly charged with ore is drawn off, and allowed to subside quietly, whereupon the "concentrate" will settle out and the "flotation oil" decanted from the surface and used over again. The gangue upon being freed from the ore, is run to the dump, and the concentrate is smelted in the usual manner.

The flotation oils may be classified as "frothers" and "collectors," the former being largely responsible for the formation of the froth, and the latter for holding the particles of ore in suspension. The "frothers" or "frothing oils" include commercial pine-oil (obtained from a destructive distillation of coniferous woods); crude hardwood tar or "pyroligneous acid" or "wood oil" obtained in the destructive distillation of maple, birch, beech, etc.; turpentine; various "essential oils"; coal-tar creosote, carbolic or "middle oil," etc. The "collectors" or "collecting oils" include crude petroleum, liquid residual oil, heavy mineral oil distillates, coke-oven tar, water-gas tar, etc. The frothing and collecting oils are mixed together in proportions depending upon the character, composition and nature of the ore to be treated. For a very "slimy" ore consisting of fine particles, a larger amount of collecting oil will be required, whereas for granular ores a higher proportion of frothing oil will be needed. A flotation oil extentively used consists of 95

per cent crude coal tar and 5 per cent of pine oil. Another mixture which is claimed to give good results contains: pine oil 10 per cent, coal-tar creosote 80 per cent, and coal tar 10 per cent. Others have suggested the use of hardwood creosote 40 per cent, coal-tar creosote 50 per cent, and coal tar 10 per cent;[245] likewise rosin pitch, phenol, alkali, acetone and glue dissolved in water.[246] The quantity of flotation oil varies from a fraction of a per cent to 3½ per cent by weight of the mineral treated. The basic U. S. patent on the flotation process calls for less than 1 per cent by weight of oil. The addition of such chemicals as caustic soda, and in some cases sulfuric acid or other agents, seems to increase the efficiency of the flotation oil and to decrease the time of treatment.

The frother systems may be classed into the mechanical, pneumatic and vacuum. In the mechanical process air is beaten into the mass; in the pneumatic method bubbles of compressed air or other gas are blown into the mixture; in the vacuum system the water is first charged with gas at atmospheric pressure and bubbles released by subjecting the mixture to reduced pressure. Mechanical froths are more permanent than those produced by pneumatic means, but the former effect a cleaner separation of coarse mineral particles, whereas the latter are better adapted for slimes. Increasing the quantity of air bubbles in the pulp, permits using less "flotation oil," also a more viscous oil, derived for example from gas-works coal tar.

In general, the following considerations should be carefully observed:

(1) The pulp should be made as thick as possible.
(2) The proportion of frothing oil to collecting oil should be carefully worked out. The more dilute the pulp or the finer the mineral particles, the larger will be the proportion of frothing oil required.
(3) The greater the aeration, the smaller will be the proportion of oil necessary.

Waterproofing Compounds for Portland-cement Mortar and Concrete. The method of incorporating various substances with Portland-cement mortar or concrete in the course of their preparation is known as the "integral" system, according to which the

waterproofing medium is mixed *throughout* the Portland-cement mortar or mass of concrete. This term does not include the application of paints or other coatings to the *surface* of mortar or concrete after it has set.

Innumerable materials have been exploited for the integral waterproofing of Portland-cement mortar and concrete including: [247]

(1) Inert fillers such as finely divided clay, infusorial earth, fuller's earth, silica, talc, etc.

(2) Active mineral fillers such as hydrated lime, aluminium hydroxide and other inorganic hydroxides, which undergo a chemical change during the setting of the cement.

(3) Water-soluble mineral salts, such as calcium chloride, sodium silicate, sodium fluorosilicate.

(4) Soap compounds including, (*a*) soluble soaps composed of sodium, potassium and ammonium combinations of animal or vegetable oils and fats, also resins; (*b*) insoluble soaps composed of calcium, magnesium, aluminium, iron, zinc, lead and other metals, combined with animal or vegetable fats and oils, or resins.

(5) Bituminous materials. We are concerned with this last group only, as falling within the scope of this treatise. The bituminous materials may be divided into two classes, namely, those used in the pure state, and those used in an emulsified form.

Pure Bituminous Substances. Two classes of bituminous substances are included in this group, namely, liquid to semi-liquid coal-tar pitch (i.e., evaporated coal tar [248]) and liquid asphalt. The latter may consist of native asphalt,[249] residual oil,[250] semi-liquid sludge asphalt,[251] asphalt-bearing diatomaceous earth,[252] residual asphalt incorporated while melted,[253] powdered hard asphalt or coal-tar pitch,[254] acid sludge,[255] spent clay obtained after refining mineral oils,[256] calcium naphthenate,[257] etc. These may be mixed with the aggregate after first wetting down the ingredients with water. An alternate procedure consists in treating the Portland cement with the bituminous substance, by atomizing the latter with compressed air in contact with the cement, so that the particles thereof are wholly or partially coated with a thin film. This treated cement is known as "bituminized cement." [258] The addition of glycerin to the melted bituminous substance is claimed to promote its admixture with water.[259] These are mixed with the aggregate after first wetting down the cement and sand in preparing Portland-cement mor-

tar; or the cement, sand, gravel or broken stone in preparing concrete. Between 5 and 25 per cent of soft coal-tar pitch or asphalt are added, based on the weight of the dry cement used. As the pure bituminous compounds are naturally water-repellent, it requires *considerable* mixing to distribute them uniformly throughout the mortar or concrete, and especially in large-sized batches. In this connection, the author wishes to lay particular emphasis on the fact that laboratory tests should under no circumstances be taken as a criterion of the ease or thoroughness with which bituminous substances may be disseminated throughout the mixture. At low temperatures, both soft coal-tar pitch and liquid asphalt become almost solid, and under these circumstances it becomes *doubly* difficult to incorporate them. To overcome these objections, a process has been described which consists in heating the cement, aggregate and bituminous matter to 200° F., and then pouring it in place and allowing it to set.

The following specifications have been proposed for an asphaltic residual oil to be used for waterproofing concrete:

(Test 7)	Specific gravity at 77° F.....	0.930–0.945
(Test 8*a*)	Specific Engler viscosity at 50° C.....	15–30
(Test 17*a*)	Flash-point (closed tester).....Min.	150° C.

There shall be no emulsification when the oil is shaken with N/100 caustic soda.

It has also been clearly shown that the introduction of residual oil alone materially decreases the tensile and compressive strength of mortar and concrete.[260] Assuming, however, that with sufficient effort the mixture can be made uniform, there is no question that the resultant mortar or concrete is improved materially in its water-repellent properties.[261]

Bituminous Substances in Emulsified Form. Bituminous emulsions tend to overcome the foregoing disadvantages, as they are readily miscible with the water used for gauging the sand, cement and crushed stone. Instead of depending upon mechanical means for incorporating the pure bituminous materials with the aggregate, such as the grinding action to which it may be subjected during the process of mixing, bituminous emulsions are first brought into suspension by stirring them into the gauging water, which then mixes

readily with the aggregate, requiring little to no effort, even at low temperatures.

The efficiency of the bituminous emulsion depends largely upon two factors, viz.: (1) the proportion of bituminous matter carried by the emulsion, and (2) whether or not its addition will interfere with the tensile or compressive strength of the cement mixture when set.

Numerous emulsions have been proposed for this purpose including: soft coal-tar pitch or asphalt emulsified by means of plastic clay;[262] soft native asphalt or residual oil emulsified with plastic clay;[263] soft coal-tar pitch emulsified by means of sodium carbonate, sodium silicate and barium peroxide;[264] residual oils emulsified by means of inorganic hydroxides capable of forming a plastic mixture with water (e.g., calcium, iron, or aluminum hydroxides);[265] melted asphalt emulsified with Portland cement and water;[266] paraffin wax emulsified with kerosene, soap and water;[267] asphalt emulsified with plastic clay and ¼ to ½ per cent ammonia;[268] paraffin wax emulsified with slaked lime;[269] powdered coal-tar pitch or asphalt of high hardness and fusing-point dispersed with slaked lime;[270] asphalt emulsified with soap;[271] asphalt or coal tar emulsified with ammonium-sulfo-oleic acid;[272] asphalt emulsified with gelatine, glue or casein;[273] asphalt emulsified with glue or gelatine in an acid solution;[274] first adding $CaCl_2$, $AlCl_3$, Na_2SiO_3, NaOH or the like to the mortar, followed by the bituminous emulsion;[275] coal tar emulsified with caustic soda and sodium silicate;[276] residual oil emulsified with $CaCl_2$ in the presence of a dispersing agent (e.g., the sludge obtained from the Dubb's cracking process, colloidal clay, bentonite, etc.);[277] carnauba wax emulsified with a mixture of sodium and potassium stearates;[278] mixing the bituminous emulsion with sawdust;[279] using a bituminous dispersion containing a dye dissolved in alcohol;[280] etc.

Bituminous emulsions give better results than any other integral waterproofing compounds. Saponifiable oils or fats and soaps (both soluble and insoluble) weaken the cement mortar or concrete, and are not nearly as permanent on exposure to the weather, or capable of continuously resisting the action of moisture.[281]

The emulsions contain a large proportion of the active bituminous constituent, mix readily with the gauging water, and may be

used in extraordinarily large proportions (20–30 per cent of the weight of cement used) without interfering with the set and at the same time *increasing* the tensile and compressive strengths of Portland-cement mixtures at least 5–15 per cent. The manufacturers recommend 2–12 lb. of emulsion per bag of Portland cement for preparing stucco and waterproof-mortar facings, and 1–2 lb. for Portland-cement concrete. These represent the *only* bituminous waterproofing preparations known to the author which may be used under all conditions, insuring a *uniform* distribution throughout the Portland-cement mixture regardless of the temperature or the means used for preparing the mixture. At the same time they may be added in sufficient quantities to secure *absolute* waterproof properties without detracting from the strength of the structure.

Methods of Use. The integral method differs from the membrane method in that it may be used for waterproofing buildings in the course of erection, as well as for repairing the *inner* surfaces of leaky masonry already constructed. The membrane method on the other hand can *only* be used on structures in the course of erection, since it must necessarily be applied on the *outer* surfaces of foundation walls, to be subsequently covered by the earth fill. Similarly, the integral waterproofing may be readily repaired should by any chance leakage occur later due to settlement or other external causes, whereas it is extremely difficult, and in many cases impossible to repair membrane waterproofing, owing to its inaccessibility.

Integral waterproofing compounds are best adapted for use in a relatively thin layer or "facing" of Portland-cement mortar, and are much more reliable when used in this manner than when incorporated throughout the body of Portland-cement concrete, for the reason that it is a comparatively simple matter to apply a layer or facing without danger of leakage at the joinings, which is not the case with mass concrete construction.

A preparation has been described for producing a glaze on concrete, brick-work and stone surfaces, consisting of Portland cement (8 parts), mineral pigment (1 part), water (10 parts), to which is added an emulsion of liquid asphalt (20 parts), sodium carbonate (15 parts), water (60 parts) and barium peroxide (5 parts).[282] A superficial glaze may also be imparted by casting the

cement mixture in contact with a hard bituminous substance.[283]

Bituminous emulsions have also been proposed for waterproofing plaster-of-Paris,[284] also magnesium-oxy-chloride compositions (i.e., Sorel cement).[285] The incorporation of soft coal-tar pitch, or pine tar [286] is claimed to give equivalent results.

Sponge Asphalt. A product termed "sponge asphalt" is recommended for calking joints, or as an insulator against heat and cold. It is prepared by directing a current of air against a stream of the melted asphalt, under conditions which will cause the air to become entrained in the asphalt as it solidifies.[287] The same effect may be obtained by adding sodium bicarbonate or ammonium carbonate to the melted asphalt; [288] or by saturating the asphalt with a gas, cooling below its melting-point, and reducing the pressure.[289] A foam-like product may also be obtained by aerating an aqueous-bituminous dispersion, through rapid beating or violent agitation,[290] or by adding alcohol, glycerol, or soap solutions to melted asphalt.[291] Sponge asphalt may for certain purposes be incorporated in Portland-cement mixtures.[292]

Asphalt Dust and Filaments. Asphalts may be converted to the form of a "dust" by being atomized in the molten condition by a blast of air; [293] by trituration cold with water; [294] by centrifuging; [295] refrigerating the asphalt and then grinding; [296] allowing the melted asphalt to spray through a cooling chamber; [297] dripping the molten asphalt into a body of cold water; [298] dripping the melted asphalt through liquefied propane; [299] cooling the melted asphalt on a cylinder filled with cold water and reducing to flakes under laterally co-acting cylinders; [300] grinding hard asphalt with litharge or other finely divided mineral matter to prevent the particles from re-adhering; [301] or treating a ground mixture of an adhesive asphalt with a solution of calcium chloride to inhibit the particles from coalescing during transportation or storage, and subsequently adding ammonium oxalate to again render the material adhesive.[302]

Asphalt dust and powder find a field of usefulness for various purposes, including the coating of pipes, etc.; [303] the manufacture of explosives (composed of powdered residual asphalt 12 parts, saltpeter 73 parts, and sulfur 15 parts); [304] the preparation of plastics by the incorporation of a solvent; [305] etc.

Asphalt Jelly. A product of jelly-like characteristics may be prepared by heating a mixture of asphalt with 7½ to 25 per cent by weight of coal tar to 140° C., stirring in an equal weight of boiling water containing caustic potash, and finally adding 1.5 per cent of an emulsifying agent;[306] or by the addition of gelatinous sodium stearate.[307]

CHAPTER XXVIII

BITUMINOUS PAVING MATERIALS

Bituminous paving materials [1] may be classified into the following groups, depending upon the specific manner in which they are to be employed, viz.:

(1) Bituminous compositions for dust-laying.
(2) Bituminous compositions containing aggregate for surface treatment.
(3) Bituminous compositions containing aggregate for repair work and patching.
(4) Bituminous macadam pavements.
(5) Bituminous concrete pavements.
(6) Sheet-asphalt pavements.
(7) Bituminous fillers for wood, brick and stone-block pavements.
(8) Asphalt-block pavements.
(9) Bituminized wood-block pavements.
(10) Asphalt mastic floorings.
(11) Bituminous expansion joints.

Each of these will be considered separately, but before taking them up, reference will be made to the two main components of bituminous pavements, namely, bituminous binders and mineral aggregates.

Bituminous Binders. The principal bituminous materials used as binders in the construction of bituminous paving materials consist of the following:

(*a*) *Asphaltic products;* including topped semi-asphaltic and asphaltic petroleums, residual oils and pressure tars, cut-back residual asphalts, blown asphalts, native asphalts and fluxed native asphalts, and fluxed asphaltites.

(*b*) *Coal-tar products;* including gas-works coal-tar, coke-oven coal-tar and water-gas coal tar, either singly or in various combinations; likewise their corresponding pitches; likewise cut-back coal-tar pitches. In special cases coal-tar distillates (e.g., creosote oil)

TABLE LVII

Specifications for Liquid Asphaltic Paving Binders

Test No.	Description of Test	Rapid-curing [4,5]								Medium-curing [4,5]										Slow-curing [5]							
		Cut-back Asphalts								Cut-back Asphalts										Residual Oils, Pressure-tars and Soft Asphalt Cements							
		RC–1		RC–2		RC–3		RC–4		MC–1		MC–2		MC–3		MC–4		MC–5		SC–1		SC–2		SC–3		SC–4	
		Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
8*aa*	Saybolt—Furol Viscosity:																										
	at 77° F., seconds	....	....	....	....	....	....	....	....	40	150	....	....	....	....	....	....	....	....	20	150	....	....	....	....	....	....
	at 122° F., seconds	80	160	200	400	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	200	320	....	....	....	....
	at 140° F., seconds	....	....	....	....	275	400	700	1400	....	....	150	250	300	500	500	800	....	....	....	....	....	....	150	300	350	550
	at 180° F., seconds	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	170	280	....	....	....	....	....	....	....	....
17*b*	Flash-point, ° F. (Cleveland open-cup)	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	150	....	200	....	200	....	250	....
17*c*	Flash-point, ° F. (Tag. open-cup)	80	....	80	....	80	....	80	....	....	....	150	....	150	....	150	....	150	....	....	....	....	....	....	....	....	....
20*a*,	Distillation Test:																										
	Distillate; per cent total vol.—																										
	Start to 374° F.	5	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	Start to 437° F.	12	....	10	....	3	....	0.5	....	....	10	....	2	....	2	....	1	....	1	....	....	....	2	....	2	....	2
	Start to 600° F.	25	....	20	....	14	....	7	....	25	....	10	20	8	20	....	16	....	14	....	....	....	15	....	10	....	8
	Start to 680° F.	....	40	....	35	....	30	....	25	....	50	....	27	....	25	....	25	....	20	....	50	....	25	....	20	....	18
	Distillation Residue—																										
8*d*	Float test at 122° F., sec.	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	50	25	....	25	....	25	....
9*b*	Penetration at 77° F. (100 g./50 cc.)	60	120	60	120	60	120	60	120	70	300	100	300	100	300	100	300	100	300	....	....	....	....	....	....	....	....
10*a*	Ductility at 77° F., cm.	60	....	60	....	60	....	60	....	60	....	60	....	60	....	60	....	60	....	....	....	....	....	....	....	....	....
21	Solubility in CS_2, per cent.	99.5	....	99.5	....	99.5	....	99.5	....	99.5	....	99.5	....	99.5	....	99.5	....	99.5	....	99	....	99	....	99	....	99	....
25	Water, per cent by weight	....	0	....	0	....	0	....	0	....	0	....	0	....	0	....	0	....	0	....	2	....	2	....	2	....	2

Intended Uses, Liquid Asphaltic Paving Binders

Use	Type	Remarks
For Dust Laying	SC-1, SC-2	Used to allay dust on earth roads.
For Priming	MC-1	Used preparatory to the application of bituminous surfacings.
For Surface Treatment	SC-2	Used for clay and sandy roads.
	MC-1	Used for silt roads.
For Seal Coats	RC-1	Used in seal-coats or in the construction of thin bituminous carpet-courses with a light cover of suitable mineral aggregate.
For Road-mix Wearing Course:		
(*a*) Dense Aggregate	MC-2	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used under severe climatic conditions.
" "	SC-2	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used in cool weather.
" "	MC-4	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used in hot weather.
" "	SC-3	
(*b*) Open Aggregate	RC-2	Aggregate maximum size not over 1½-in., having little or no material passing No. 200 sieve; used in cool weather; also for patching purposes.
" "	RC-3	Aggregate maximum size not over 1½-in., having little or no material passing ¾-in. screen; used under normal climatic conditions, also for cold patching work.
" "	MC-3	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used under severe climatic conditions.
For Plant-mix Wearing Course:		
(*a*) Dense Aggregate	MC-3	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used under severe climatic conditions.
" "	SC-4	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve; used in dry climatic conditions.
(*b*) Open Aggregate	RC-4	Aggregate maximum size not over 1½-in., having little or no material passing ¾-in. screen.
" "	MC-4	Aggregate maximum size not over 1-in., having relatively high percentage passing No. 200 sieve.
" "	MC-5	Aggregate maximum size not over 1-in., having little or no material passing No. 200 sieve.

are used for impregnating wood blocks. The addition of rubber or chlorinated rubber [2] has also been suggested. Another expedient consists in reworking old asphalt pavements after they have worn out and incorporating a proportion thereof with the freshly prepared mixture.[3]

Table LVII embraces the specifications for liquid asphaltic paving binders, Table LVIII * the specifications for semi-solid to solid asphaltic paving binders, and Table LIX † those applicable to coal-tar binders as currently standardized in the U.S.A. These have been classified according to the various types of pavements and for the sake of convenience will hereinafter be referred to by the key designation of the particular product.

Bituminous materials are used without other additions for application as bituminous dust-laying oils, fillers for wood, brick or stone pavements and in certain forms of expansion joints; and they are used in admixture with mineral aggregate for constructing bituminous surfacings, bituminous macadam, bituminous concrete pavements, sheet asphalt pavements, asphalt-block pavements, asphalt mastic flooring and certain types of expansion joints. They are also used in combination with wood for manufacturing bituminized wood-block pavements; with felted fabric in forming certain bituminous expansion joints; and in some cases emulsified with water for preparing dust-laying oils, and bituminous cements to be used cold.

Asphalt binders for use in constructing asphalt macadam, asphaltic concrete, sheet-asphalt pavements and grout fillers for stone-block and brick pavements, also binders for paving blocks are usually designated by their penetration ranges at 77° F. (Test 9*b*). Their selection depends upon the severity of the traffic and upon the climatic conditions as stated in Table LX.[85]

The term "asphalt cement," abbreviated A.C., is applied to an asphalt suitable for direct use in highway work and includes asphalts brought to a consistency desired for a particular use, either by distillation or by mixing or fluxing with a harder or a softer asphalt, as the case may be. A hard asphalt which must be fluxed or softened to the proper grade is known as a "refined asphalt," abbreviated R.A. The softening agent is known to the paving industry as a "flux" or "flux oil." The presence of paraffin in crystallizable

* References 4 to 18 incl. appear in Table LVIII (insert).

† References 19 to 34 incl. appear in Table LIX (insert).

TABLE LVIII. SPECIFICATIONS FOR SEMI-SOLID TO SOLID ASPHALTIC PAVING BINDERS

	Key designation of Product	E		F	G	H				I				J			K			L			M			N	
		Asphalt Paving Blocks		Asphalt Macadam....60–70, 85–100, 100–120 and 120–150 Asphaltic Concrete—Coarsely Graded Type....30–40, 40–50, 50–60 and 60–70 Asphaltic Concrete—Finely Graded Type....25–30, 30–40, 40–50, 50–60 and 60–70 Sheet Asphalt Pavements....25–30, 30–40, 40–50, 50–60 and 60–70 Grout Filler for Stone-block Pavements....40–50, 50–60 and 60–70 Grout Filler for Brick Pavements (Unblown Type)....40–50																		Grout Filler for Brick Pavements (Blown Type)	
Test No.	Description of Test	10–15	15–25	25–30	30–40	40–50				50–60				60–70			85–100			100–120			120–150			30–50	
		A.S.T.M.	A.S.T.M.	A.S.T.M.	A.S.T.M.	A.S.T.M.	F.S.B.[11]			A.S.T.M.	F.S.B.[11]			A.S.T.M.	F.S.B.[11]		A.S.T.M.	F.S.B.[11]		A.S.T.M.	F.S.B.[11]		A.S.T.M.	F.S.B.[11]		A.S.T.M.	F.S.B.[18]
		(6)	(7)	(8)	(9)	(10)	AP–7–25	AB–7–25	AT–7–25	(12)	AP–6–25	AB–6–25	AT–6–25	(13)	AP–5–25	AB–5–25	(14)	AP–3–25	AB–3–25	(15)	AP–2–25	AB–2–25	(16)	AP–1–25	AB–1–25	(17)	PAF–1–25
		Petrol. Asphalt	Petrol. Asphalt	Petrol. Asphalt	Petrol. Asphalt	Petroleum Asphalt		Fluxed Natural	Fluxed Trind.	Petroleum Asphalt		Fluxed Natural	Fluxed Trind.	Petroleum Asphalt		Fluxed Natural	Petroleum Asphalt		Fluxed Natural	Petroleum Asphalt		Fluxed Natural	Petroleum Asphalt		Fluxed Natural	Petroleum Asphalt	
2a	Appearance to the eye	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.	Homog.
7	Specific gravity at 77/77° F.....Min.	.....	.....	.....	.....	.....	1.010	1.055–1.075	1.210–1.270	.....	1.010	1.050–1.070	1.200–1.250	.....	1.010	1.050–1.070	.....	1.000	1.050–1.070	.....	1.000	1.040–1.060	.....	1.000	1.025–1.050	.....	.....
9b	Penetration at 115° F.....Max.	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	110	110
	Penetration at 77° F.....Max.	10–15	15–25	25–30	30–40	40–50	40–50	40–50	40–50	50–60	50–60	50–60	50–60	60–70	60–70	60–70	85–100	85–100	85–100	100–120	100–120	100–120	120–150	120–150	120–150	30–50	30–50
	Penetration at 32° F.....Min.	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	10	10
10a	Ductility at 77° F., cm.....Min.	5–15	5–20	15	25	30	40	40	40	30	40	40	40	30	40	40	30	.....	.....	30	.....	.....	30	.....	.....	3	2
15b	Fusing-point ° F. (R. and B. method).....	.....	.....	.....	.....	.....	113–149	113–131	122–140	.....	104–140	113–131	113–131	.....	104–140	113–131	.....	104–140	104–122	.....	95–131	104–122	.....	95–131	95–113	149–230	149–230
16	Volatile at 325° F., 5 hours, per cent.....Max.	1	1	2	2	2	1	3	3	2	1	3	3	2	1	3	2	1	3	2	1	3	2	1	3	1	1
9b	Resid. at 325° F., pen. at 77° F. vs. orig. pen., per cent..Min.	50	50	60	60	60	60	50	50	60	60	50	50	60	60	50	60	60	50	60	60	50	60	60	50	60	60
8d	Resid. at 325° F., float test at 122° F., sec.....Min.																										
17b	Flash-point ° F. (open cup).....Min.	392	392	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	347	392	392
21	Soluble in carbon disulfide, per cent.....Min.	.....	.....	.....	.....	.....	99.5	94.0	65.0	.....	99.5	94.0	68.0	.....	99.5	94.5	.....	99.5	95.0	.....	99 5	95.0	.....	99.5	95.0	.....	99 0
21	Non-mineral matter insoluble, per cent.....Max.	.....	.....	.....	.....	.....	0.2	.....	.....	.....	0.2	.....	.....	.....	0.2	.....	.....	0.2	.....	.....	0.2	.....	.....	0.2	.....	.....	.....
21	Mineral matter.....	.....	.....	.....	.....	.....	.....	2.5–4.0	22–32	.....	.....	2.5–4.0	20–30	.....	.....	2.0–3.5	.....	.....	1.5–3.0	.....	.....	1.5–3.0	.....	.....	1.5–2.5	.....	.....
22	Soluble in carbon tetrachloride, per cent.....Min.	99	99	99	99	99	.....	.....	.....	99	.....	.....	.....	99	.....	.....	99	.....	.....	99	.....	.....	99	.....	.....	99	99
25	Water, per cent.....	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

To face page 570.

form in excess of 4 to 5 per cent is likely to reduce the adhesiveness and cohesiveness of asphaltic cements, and hence is considered objectionable.[36]

Coal-tar binders are generally distinguished by their carbon con-

TABLE LX

ASPHALTIC BINDERS FOR VARIOUS TRAFFIC AND CLIMATIC CONDITIONS, BASED ON THEIR PENETRATION RANGES AT 77° F. (TEST 9*b*)

	Climatic Conditions		
	Low Temperatures (Northern U.S.)	Moderate Temperatures (Middle U. S.)	High Temperatures (Southern U.S.)
Asphalt Macadam (Base and Surface Courses):			
Light traffic	120–150	100–120	85–100
Moderate traffic	100–120	85–100	60–70
Heavy traffic	85–100	85–100	60–70
Asphalt Concrete—Coarsely-graded Type (Base and Surface Courses):			
Light traffic	60–70	60–70	50–60
Moderate traffic	60–70	60–70	40–50
Heavy traffic	50–60	50–60	30–40
Asphalt Concrete—Finely-graded Type (Base and Surface Courses); also Sheet Asphalt (Binder and Surface Courses):			
Light traffic	60–70	50–60	40–50
Moderate traffic	50–60	50–60	30–40
Heavy traffic	40–50	40–50	25–30
Grout Filler for Stone-block Pavements:			
All traffic conditions	60–70	50–60	40–50
Grout Filler for Brick Pavements:			
All traffic conditions (unblown type)	40–50	40–50	40–50
All traffic conditions (blown type)	30–50	30–50	30–50
Asphalt Paving Blocks:			
Light and moderate traffic	15–25	15–25	10–15
Heavy traffic	15–25	10–15	10–15

tent (i.e., free carbon). There is still a difference of opinion as to whether a binder of high-carbon or low-carbon content is more suitable, and there are proponents of both types.

Cut-back products are used in highway work for the following purposes:[37]

(1) Priming road surfaces prior to surface treatment, seal-coating or constructing a wearing-course.

(2) Surface treatment to produce a carpet or seal-coat.

(3) Preparation of mixtures with aggregate to produce a wearing course of substantial thickness for cold application, also for cold-patching.

(4) Preparation of mixtures with aggregate to produce a base-course for cold application.

Bituminous Emulsions. Emulsions[38] are prepared in accordance with the methods outlined in a preceding chapter. The dispersing agents in common use consist of soaps, used either alone or in combination with other agents. The bituminous constituent is usually asphalt, and coal-tar products are rarely used, at least in the United States. The emulsion is customarily brought about by means of a high-speed disintegrator or so-called colloid mill.

The conditions favoring the formation of emulsions and their stabilization during storage are reversed when the emulsion is used on road surfaces. Fine subdivision is destroyed by the separation of water, causing an increase in concentration until its stability is destroyed, and at the same time the electrical charges are altered by contact with the mineral aggregate, resulting in its agglomeration. The principal object of a road emulsion is the distribution of the bituminous substance over and into the road surface by means of an aqueous vehicle, which deposits its burden and then disappears. The following characteristics are desirable:[39] (1) The bituminous constituents must be of high quality; (2) the dispersion must be stable at high as well as low temperatures, with very little agglomeration, even after months of standing; (3) the dispersion should have the proper degree of fluidity, so that it may be easily applied, and at the same time contain 56–60 per cent by weight of bituminous constituents; (4) the dispersion after setting must be impervious to water and form a stable coating at all temperatures, or in other words, the dispersing agent must be present in the smallest possible amount, partly to prevent re-emulsification under traffic, and partly to avoid reducing the adhesive properties of the base, in relation to the mineral matter of the road surface.

Road emulsions have advantages over non-emulsified products, in that they may be applied during damp weather, and that the

quantity applied is controlled automatically, because any excess will drain off through the aggregate into the subgrade, leaving the proper amount to remain in the interstices of the former. On the other hand, emulsions have the disadvantage of carrying a certain amount of water in its composition, which has no value from the standpoint of the finished pavement and accordingly necessitates the purchaser paying freight on this inactive ingredient.

The following specifications have been proposed for different types of emulsions for road work:[40]

I. Open-graded coarse-aggregate plant mixtures.
II. Open-graded coarse-aggregate for road-mix construction and resurfacing.
III. For summer use in heavy premix bituminous concrete mixtures for repair work.
IV. For winter use in heavy premix bituminous concrete mixtures for repair work.
V. For quick-setting penetration macadam and surface treatment work.

Types I, II, III and V emulsions shall be homogeneous and shall show no separation of asphalt at the time of use, provided that separation has not been caused by freezing after delivery, and provided further that this requirement shall not apply if the material is held more than 30 days after delivery.

Type IV emulsion shall be homogeneous and shall show no separation of asphalt at the time of use, provided that this requirement shall not apply if the material is held more than 30 days after delivery.

The emulsions shall conform to the tests included in Table LXI.

To hasten the breaking of bituminous emulsions, it has been proposed to previously moisten the mineral aggregate with sodium silicate;[41] or with an alkali solution;[42] or a solution of metallic salts (e.g., lead acetate);[43] or with a solution of suitable electrolytes (e.g., $CaCl_2$, NaCl, Na_2SO_4, etc.). An alternate procedure consists in first mixing the aggregate with an oil emulsion;[44] or with a small amount of diluted bituminous emulsion;[45] or with a mixture of bituminous emulsion containing mineral fillers;[46] or with a mixture of the emulsion and powdered hard asphalt.[47]

TABLE LXI. SPECIFICATIONS FOR EMULSIFIED ASPHALTS

Test No.	Description	I	II	III	IV	V
	Emulsion in Original Condition:					
92*b*	Sieve Test.....Max. %	0.10* 0.20†	0.10*	0.10*	0.10*	0.10*
			0.20†	0.20†	0.20†	0.20†
92*c*	Settlement, 5 days .....Max. %	5	5	....	....	3
93	Saybolt Furol Viscosity Min.	100	20	....	....	20
	60 ml. at 77° F. secs. Max.	700	100	....	....	100
94*a*	Demulsibility ..... Min. %	....	....	....	....	50†§
	Max. %	30‡	30‡	....	....	60*§
94*b*	Freezing Test, 3 cycles.....	....	....	....	††‖	....
94*c*	Coating Test.....	¶	¶	¶	¶	....
95	Miscibility with water.....	**	**	**	**	....
98	Distillation residue .... Min. %	60	55	65	65	55
	Max. %	65	60	....	....	60
	Residue from Distillation Test:					
7*a*	Specific gravity at 77° F. Min.	1.00	1.00	1.00	1.00	1.00
9*b*	Penetration at 77° F.... Min.	100	100	100	125	100
	Max.	200	200	200	225	200
10	Ductility at 77° F., cm...Min.	40	40	40	40	40
21	Soluble in carbon disulf...Min. %	95††	95††	95††	95	95††
21	Ash.....Max. %	2	2	2	2	2

Note: Tests refer to same figures being included in *both* the A.S.T.M. and F.S.B. specifications, unless otherwise noted.

* A.S.T.M Specifications only.
† F S.B. Specifications only.
‡ Using 50 ml. of 0 10 N $CaCl_2$ solution.
§ Using 35 ml. of 0 02 N. $CaCl_2$ solution.
‖ Homogeneous.
¶ Shall show no appreciable separation when mixed with washed, dry, clean stone for 3 min. and shall coat the stone thoroughly.
** Shall show no appreciable coagulation in 2 hrs.
†† For native asphalts. For petroleum asphalts 97.5 per cent. minimum (A.S.T.M. Specifications).

Mineral Aggregates. In the case of constructional materials, the term "aggregate" has been defined [48] as inert material which when bound together into a conglomerated mass by a matrix, forms concretes, mastic, mortar, plaster, etc. The types of mineral aggregates used in the various classes of paving construction, based upon their granularmetric analysis (Test 60) are given in Table LXII.* For the sake of convenience, these will hereinafter be referred to by the key designation of the particular aggregate.

Table LXIII * indicates the particular aggregates (referred to by key designation) recommended for use in the various types of pavements, based on U. S. A. practice. The coarse and intermediate aggregates shall be clean, free from dust and an excess of flat or

* References 49 to 72 incl. appear in Tables LXII (insert) and LXIII.

elongated fragments. Gravel shall be composed of sound, hard, durable pebbles, free from clay or surface coatings of any character. The fine aggregate shall consist of broken stone screenings or sand, composed of clean, hard, durable grains, free from clay, loam and other foreign matter. The filler or dust shall consist of thoroughly dry limestone dust, dolomite dust, or Portland cement.

The "percent of wear," [73] "toughness" [74] and "weight per cubic foot" [75] shall be as follows:

	Broken Stone or Gravel		Broken Slag
	Percent of Wear	Toughness	Lbs. per cu. ft.
Bituminous Macadam:			
Base Course	< 6–8	> 6	> 65–70
Surface Course	< 6	> 6	> 70
Bituminous Concrete:			
Base Course	< 6–8	> 6	> 70
Surface Course { Coarsely-graded	< 5–6	> 6	> 70
Surface Course { Finely-graded	< 6–7	> 6	> 70
Sheet Asphalt:			
Binder Course	< 6	> 6	> 70

BITUMINOUS COMPOSITIONS FOR DUST-LAYING (COLD APPLICATION)

Dust-laying compositions are also designated "dust preventatives" or "dust palliatives." They are usually liquid at room temperature and are adapted for use without heating, on earth, gravel or macadam roads, for temporarily resisting the formation and dispersion of dust under traffic conditions. Dust-laying oils may either be used as such, or in an emulsified state with water, and are applied to the road, after removing the loose particles of dust, by any suitable form of sprinkling or spraying device, preferably under pressure. The function of the dust-laying oil is twofold, namely, to prevent the dispersion of dust already formed, and retard the formation of additional particles under the attrition of traffic. The saturating, *adhesive* or *binding* properties of the oil accomplish these results. At best, the effect of dust-laying oils is temporary, and they must accordingly be renewed from time to time, usually twice during the first season, and once each year thereafter, assuming that the traffic conditions are not abnormally severe.

Bituminous Emulsions. Emulsions, on account of their greater

TABLE LXIII

AGGREGATES USED IN VARIOUS TYPES OF PAVEMENTS

Note:—Roman Numerals designate alternates	Federal Specification Board, Wash., D. C.	American Society for Testing Materials	Asphalt Association
BITUMINOUS MACADAM			
Base Course			
(*a*) *First Layer*			
Coarse Aggregate	B [55]	A [50, 51, 52]	A [54]
(*b*) *Second Layer*			
Coarse Aggregate	E [55]	C [50, 51, 52]	C [54]
Intermediate Aggregate (*)	J [55]	H [50, 51, 52]	J [54]
Surface Course			
Coarse Aggregate	I: B [55] II: E [55]	I: H [49, 50, 52] II: C [49, 50, 52]	C [54]
Intermediate Aggregate	J [55]	H [49, 50, 52]	J [54]
Seal Coat			
Intermediate Aggregate	M_3 [55]	L_1 [49, 50, 52]	L_1 [54]
BITUMINOUS CONCRETE			
Base Course			
I: Open Mixture			
Coarse Aggregate	E [60]	C [56]	.....
II: Closed Mixture			
Coarse or Intermed. Aggregate	I: F [60] II: K [60]	I: D_1 [56, 57]; D_2 [59] II: I_1 [52, 57]; I_2 [59]	I: D_1 [54] II: G [54] Mixture
Fine Aggregate (sand)	.....	.....	P_1 [54]
Surface Course			
I: Coarsely-graded Type			
Mixture of: Intermed. Aggregate	K [60]	I_1 [52, 61, 62]	I_1 [54]
Mixture of: Fine Aggregate (sand)	Q_1 [68]	Q_1 [69]	P_1 [54]
Mixture of: Filler	S_1 [72]	S_1 [71]	S_2 [54]
II: Finely-graded Type			
Mixture of: Intermed. Aggregate	N [60]	I: L_1 [62] II: M_2 [52, 65]	I: L_2 [54] II: M_2 [54]
Mixture of: Fine Aggregate (sand)	Q_1 [68]	Q_1 [69]	Q_2 [54]
Mixture of: Filler	S_1 [72]	S_1 [71]	S_2 [54]
Seal Coat (for Coarsely-graded Type)			
Intermediate Aggregate	N [60]	I: L_1 [52, 61] II: M_1 [62]	M_1 [54]
SHEET ASPHALT PAVEMENT			
Binder Course			
Mixture of: Intermediate Aggregate	K [63]	I_1 [52]	I_3 [54]
Mixture of: Fine Aggregate (sand)	P_2 [68]	Q_1 [69]	P_1 [54]
Surface Course			
Mixture of: Fine Aggregate (sand)	Q_1 [68]	Q_1 [69]	Q_2 [54]
Mixture of: Filler	S_1 [72]	S_1 [71]	S_2 [54]

* Intermediate Aggregate used: (1) if a finely-graded Bituminous Concrete surface-course, or (2) if a Sheet-asphalt surface-course is to be laid over the Bituminous Macadam base-course.

fluidity, have the advantage of being applied more cheaply than untreated oils or tars, thus enabling them to be spread on the road without the use of a special form of apparatus. In addition, they are absorbed more rapidly by the road surface, and obviate the necessity of interfering with traffic while applied, or the annoyance of having the oil tracked about by pedestrians, as often proves the case when crude petroleums or tars are used.

Non-Emulsified Products. Non-emulsified oils and tars are usually less expensive in the long run. They are generally applied cold, but in certain cases, and particularly during cold weather, better results may be obtained by sprinkling them on the road in a heated state. The best practice necessitates using from 0.3 to 0.7 gal. per sq. yd., depending upon the nature of the road, the quantity of dust *in situ,* and whether or not the road has been oiled previously. On a road oiled at regular intervals, 0.1 to 0.2 gal. per sq. yd. is sufficient after the initial application. The surface should then be sprinkled lightly with sand, using a cubic yard for every 75 to 125 sq. yd. of surface, to prevent the oil being tracked about by pedestrians and vehicles. A longer period should be given the oil to soak into gravel or macadam roads since they are denser and less porous than dirt roads. For the same reason, the oil must be applied with great care to provide a uniform distribution and to prevent it accumulating in "pools."

The following products are generally employed: topped petroleums of semi-asphaltic and asphaltic nature, residual oils and pressure tars, cut-back residual asphalts, coal tars (including gas-works, coke-oven and water-gas coal-tars), and cut-back coal-tar pitches. It is still a mooted question as to whether topped petroleums, residual oils and pressure tars derived from asphaltic, semi-asphaltic or non-asphaltic petroleum give the best results in service, and much may be said on both sides of this question.[76] Cut-back products have the advantage of setting up rapidly when applied to the road. The specifications for dust-laying compositions of asphaltic and coal-tar types respectively are given in Tables LVII and LIX.

General Considerations. The following precautions should be followed in applying the dust-laying composition: All the loose dust should be removed from the road before applying the oil, which should be spread on a warm, sunny day on to the dry road surface.

The oil should be allowed to soak into the road thoroughly before reopening it to traffic. Bituminous emulsions should be applied under similar conditions, except that the road need not necessarily be dry, since they will bond equally well with moist surfaces.

BITUMINOUS SURFACINGS

A bituminous surfacing consists of a layer of appreciable thickness constructed on top of a newly prepared or an old roadway (e.g., silt, clay, sand, gravel, macadam, concrete, brick, block, or old bituminous pavements), by the application of one or more coats of bituminous material, interposed with stone-chips, sand or gravel, the purpose of which is to fill the voids of the foundation and produce a smooth and uniform wearing surface, sometimes termed a "carpet", "carpeting coat", or "seal coat". Other objectives include the elimination of dust formation by attrition, and to provide a somewhat elastic cushion which makes the pavement less noisy and increases the comfort of those who travel over it. Bituminous surface treatments have been in vogue in the United States, since 1894, when they were first introduced at Santa Barbara, California.

The following constitute the principal classes of bituminous surfacings:

	Mixture Prepared	Binder Introduced	Mixture Applied to Road	Compacted by Means of
Surface treatment	In situ	Cold	.....	Traffic
Seal coats	In situ	Heated	.....	Rolling
Road-mix Wearing Course:				
Dense aggregate	In situ	Heated	.....	Traffic
Open aggregate	In situ	Heated	.....	Rolling
Plant-mix Wearing Course:				
Dense aggregate	At plant	Heated	Cold	Traffic
Open aggregate	At plant	Heated	Cold	Rolling

In the United States, either asphaltic or coal-tar products are employed as binders, whereas in Great Britain and Europe, mixtures of residual asphalts fluxed with tars are used extensively. In Germany, coal tars cut back with a volatile coal-tar distillate, are known as "cold tars".[77]

The following methods of application have been proposed involving the use of asphaltic binders:[78]

SURFACE TREATMENT

Wearing course not exceeding 1-in. in thickness.

A. Silt Roads:

1. Surface bladed and dragged.
2. First application: MC-1 0.5 gal. per sq. yd. applied cold, without cover-coat.
3. Second application several days later: MC-1 0.25 gal. per sq. yd. applied cold without cover-coat.
4. Third application several weeks later: MC-1 0.25 gal. per sq. yd. applied cold, without cover-coat.
5. Retreat when necessary, with MC-1 0.25 gal. per sq. yd. applied cold without cover-coat.

B. Clay Roads:

(*a*) *Non-capillary types of clay* (i. e., "blotter" or "mulch" treatment):

1. Surface bladed and dragged.
2. Prime with MC-1 0.3 gal. per sq. yd. applied cold, without cover-coat.
3. Apply SC-2 0.3 gal. per sq. yd. applied cold; cover with gravelly sand 50 lbs. per sq. yd.; blade and drag until thoroughly mixed; and subject to compacting by traffic.
4. Retreat when necessary, with SC-2 0.2 to 0.3 gal. per sq. yd. applied cold; cover with gravelly sand 10–20 lbs. per sq. yd.; reblade and drag until thoroughly mixed; and permit compacting by traffic.

(*b*) *Highly-capillary types of clay*:

1. Surface smoothed and consolidated as densely as possible.
2. Apply asphalt cement (250 penetration at 77° F.) 0.5 to 0.7 gal. per sq. yd.; cover with earth layer 12-in. thick; smooth and consolidate as densely as possible; and permit compacting by traffic.
3. Follow as in 2, 3 and 4 of preceding.

C. Sandy Roads:

1. Surface bladed to a depth of 1-in. and dragged, retaining the material to one side of the road.
2. Apply SC-2 cold 0.6 to 0.8 gal. per sq. yd.; cover with the surfacing previously removed and allow same to be consolidated by traffic.
3. At the end of several months repeat 1 and 2.
4. Retreat when necessary as in 1 and 2.

SEAL-COATS

A. To Loose Surfaces:

1. Surface bladed and dragged.

2. Prime with MC-1 0.5 gal. per sq. yd. applied cold, without cover-coat.

3. Apply a seal-coat, consisting either of:

(*a*) RC-1 0.25 gal. per sq. yd. applied at 125–140° F.; cover with sandy gravel, crushed slag, or crushed rock (ranging from ¼ to ¾-in.) 25-lbs. per sq. yd.; broom-drag and roll until smooth.

(*b*) Asphalt-cement (100–350 penetration at 77° F.) 0.3 gal. per sq. yd. applied at 300° F.; cover with crushed aggregate (ranging from ¼ to ¾-in.) 30-lbs. per sq. yd.; broom-drag and roll until smooth and compacted.

(*c*) Emulsified asphalt 0.15 to 0.20 gal. per sq. yd.; cover with crushed aggregate (ranging from ¾ to 1¼-in.) 40-lbs. per sq. yd.; smooth and roll until compacted. Apply a second coat of emulsified asphalt 0.25 to 0.40 gal. per sq. yd.; cover with crushed aggregate (ranging from ¼ to ¾-in.) 20-lbs. per sq. yd.; smooth and roll until compacted. Apply with a third coat of emulsified asphalt 0.35 to 0.40 gal. per sq. yd.; cover with "birdseye" screenings (3-mesh) 15-lbs. per sq. yd.; smooth and roll until compacted.

4. Retreat when necessary as in 1, 2 and 3.

B. To Bonded Surfaces:

1. Prime with MC-1 0.25 to 0.3 gal. per sq. yd. applied cold, without cover-coat.

2. Apply a seal-coat, consisting either of:

(*a*) RC-1 0.25 to 0.35 gal. per sq. yd. applied at 125–140° F.; cover with crushed aggregate (ranging from ¼ to 1-in.) 25-lbs. per sq. yd.; broom-drag and roll until smooth and compacted.

(*b*) Asphalt-cement (250 penetration at 77° F.) 0.4 gal. per sq. yd. applied at 300° F.; cover with crushed aggregate (ranging from ¾ to 1¾-in.) 40-lbs. per sq. yd.; broom-drag and roll until smooth and compacted.

(*c*) Emulsified asphalt as in A-3-(c).

3. Retreat when necessary, as in 1 and 2.

C. To Old Bituminous Surfaces:

This involves the surface treatment or retreatment of old bituminous pavements, such as previously treated macadam or penetration macadam surfaces.[79]

1. Patch depressions with not less than ¾-in. of a mixture of RC-2 12 to 15 gal. per ton of aggregate (ranging from ¾ to 1¼-in., or else from ¼ to ¾-in.).

2. Apply an evener course, consisting either of:

(*a*) RC-1 0.25 gal. per sq. yd. applied at 125–140° F.; cover with crushed aggregate (ranging from ¼ to ¾-in.) 25-lbs. per sq. yd.; broom-drag, blade and roll until smooth and well compacted; then open to traffic for several days.

(*b*) Emulsified asphalt as in A-3-(c).

3. Apply a seal-coat, consisting either of:

(*a*) RC-1 0.15 to 0.25 gal. per sq. yd. at 80–125° F.; cover with broken-stone, mine-tailings or slag (passing ¾-in. screen 100 per cent, passing ½-in. screen not less than 95 per cent, passing ¼-in. screen not more than 10 per cent) 15 to 25-lbs. per sq. yd.; broom-drag, blade and roll with a power-driven roller weighing not less than 5-tons until well compacted and smooth.

(*b*) Asphalt-cement (100–350 penetration at 77° F.) 0.25 to 0.4 gal. per sq. yd. at 300° F.; cover with crushed aggregate (ranging from ¾ to 1¾-in.) 30–40 lbs. per sq. yd.; broom-drag, blade and roll with a power-driven roller weighing not less than 5-tons until well compacted and smooth.

(*c*) Emulsified asphalt as in A-3-(c).

4. Light retreatments when necessary, as in 3.

D. To Non-Bituminous Surfaces:

This involves the surface treatment of Portland-cement concrete, block or brick pavements.

1. Remove all dust and patch depressions with not less than ¾-in. of a mixture of RC-2 12 to 15 gal. per ton of aggregate (ranging from ¾ to 1¼-in., or else from ¼ to ¾-in.).

2. Prime with either MC-1 applied cold, or with emulsified asphalt 0.12 to 0.25 gal. per sq. yd. without cover-coat.

3. Follow as in 2 and 3 of preceding section.

4. Light retreatments when necessary, as in 3 of preceding section.

ROAD-MIX WEARING COURSE

Wearing course not less than 1 nor more than 4-in. thick.[80]

A. Open Aggregate ("Macadam Type"):[81]

1. The foundation, consisting of well compacted roads 6 to 8-in. thick (e. g. water-bound macadam or other well bonded roads) must be leveled and swept clean.

2. Prime with MC-1 applied with a pressure distributor at 50 to 150° F. at a uniform rate of from 0.3 to 0.5 gal. per sq. yd.

3. Spread coarse aggregate (broken stone, mine tailings, crushed gravel or slag of the following dimensions: 1½–½-in. coarse aggregate of which 95–100 per cent passes 1½-in. screen, 25–75 per cent passes 1-in. screen and 0–10 per cent passes ½-in. screen) over the primed foundation in uniformly loose layer 2½-in. thick.

4. Apply asphalt binder RC-3 by means of a pressure distributor at 100–175° F. at a uniform rate of 0.3 to 0.5 gal. per sq. yd. (depending upon the type of aggregate) and blade thoroughly with the aggregate. Apply a second coat of binder RC-3 at a uniform rate of 0.3 to 0.5 gal. per sq. yd. and again blade. Compress with a power-driven roller weighing not less than 10-tons with a minimum weight of 300-lbs. per inch width of rear wheel.

5. Not less than a week later, spread fine aggregate (broken stone, mine tailings, crushed gravel or slag of the following dimensions: ¾–¼ in. fine aggregate of which 95–100 per cent passes ¾-in. screen and 0–10 per cent passes ¼-in. screen) at a uniform rate of 15–18 lbs. per sq. yd. and lightly broom-drag. Apply a seal-coat of asphalt binder RC-3 at the rate of 0.2–0.3 gal. per sq. yd., broom-drag and roll until smooth.

B. Dense Aggregate ("Graded Type"):[82]

1. The foundation, consisting of loosely compacted roads 6 to 8-in. thick (e. g. old gravel or traffic-bound stone roads), must be leveled and swept clean.

2. Prime with MC-1 applied with a pressure distributor at 50 to 150° F. at a uniform rate of 0.3 to 0.5 gal. per sq. yd.

3. Spread mixed aggregate (broken stone, crushed or uncrushed gravel, sand, stone screenings and mineral dust of the following dimensions: passing 1-in. screen 100 per cent, total passing ¼-in. screen 50–70 per cent, total passing 10-mesh sieve 35–60 per cent, passing 200-mesh sieve 5–14 per cent; of which that portion passing the 10-mesh sieve shall contain not more than 20 per cent moisture and show a lineal shrinkage of not more than 5 per cent) over the primed foundation in a layer to produce a finished wearing course of specified thickness.

4. Apply asphalt binder MC-2 by means of a pressure distributor at 160–200° F. in successive treatments at the rate of ½-gal. per sq. yd. each so as to produce a final mixture containing 4–7 per cent of asphalt. Blade after each application of the binder until all free asphaltic material has been mixed into the aggregate. Compress with a power-driven roller weighing 5–8 tons.

5. Apply a seal-coat of asphaltic binder MC-2 at the rate of 0.2 to 0.3 gal. per sq. yd., broom-drag and roll until smooth.

PLANT-MIX WEARING COURSE

A. Open Aggregate ("Macadam Type"):

Follow the same procedure as described under "Road-Mix Wearing Course," except that the coarse aggregate and asphalt binder RC-3 (sections 3 and 4) are mixed together at the plant instead of on the road.

B. Dense Aggregate ("Graded Type"):

Follow the same procedure as described under "Road-Mix Wearing Course," except that the mixed aggregate and asphalt binder MC-2 (sections 3 and 4) are mixed together at the plant instead of on the road.

BITUMINOUS MACADAM PAVEMENTS [83]

These are laid in a manner similar to ordinary macadam except that the aggregate is bound together by bituminous matter which is applied by the penetration method after the aggregate has been spread in place.[84] The wearing course is then finished off with a surface treatment of bituminous binder, which when covered with stone chips produces a "seal coat." This form of pavement is also known as "road-mix," "mixed-in-place," "penetration-mix," "asphalt macadam," "asphalt broken stone," "bituminous gravel," "bituminous broken stone," etc., depending upon the particular materials employed in its construction.

Foundation. The thicker and firmer the foundation, the more durable the pavement will be. Either a macadam or a Telford or a well-drained gravel foundation is recommended. Old brick, stone block or concrete foundations will also answer. Sand or gravel roads are frequently too soft to give good results. If the foundation is worn or rutted or filled with holes, it should be levelled and rolled before the surface course is applied. In constructing new roads the foundation may consist of aggregate A.

Base Course. This shall be constructed in the following manner, when asphaltic binders are used: [85]

1. The foundation (i.e., sub-base) should be swept clean.
2. Spread 2½–1¼ in. coarse aggregate (broken stone, mine tailings or slag of which 95–100 per cent passes 2½-in. screen, 25–75 per cent passes 2-in. screen and 0–5 per cent passes 1¼-in.

screen) in a uniformly loose layer of a thickness to provide a finished layer not less than 3-in. thick. Compress with a power-driven roller weighing not less than 10-tons with a minimum weight of 300-lbs, per inch width of rear wheel.

3. Apply asphalt paving binder J, K, L or M (dependent upon the climatic conditions) by means of a pressure distributor at 275–350° F. at a uniform rate of 1.25 gal. (± 0.1 gal.) per sq. yd.

4. Immediately after applying the binder, and while it is still warm, spread a light sprinkling of ¾–¼-in. fine aggregate (broken stone, mine tailings or slag, of which 95–100 per cent passes ¾-in. screen, 25–75 per cent passes ½-in. screen and 0–5 per cent passes ¼-in screen) and continue rolling until smooth.

5. A second application of asphalt binder at a rate of 0.4 gal. (± 0.1 gal.) per sq. yd., followed by a sprinkling of ¾–¼-in. fine aggregate, completed by broom-dragging and rolling, until smooth and free from depressions.

Surface Course. When asphaltic binders are employed, the surface course shall be constructed as follows:[86]

1. The base course should be swept clean.

2. Spread 2½–1¼-in. coarse aggregate (broken stone, mine tailings or slag, of which 95–100 per cent passes 2½-in. screen, 25–75 per cent passes 2-in. screen and 0–5 per cent passes 1¼-in. screen) in a uniformly loose layer to provide a finished layer not less than 2½-in. thick. Compress with a power-driven roller weighing not less than 10-tons, with a minimum weight of 300-lbs. per inch width of rear wheel.

3. Apply asphalt paving binder J, K, L or M (depending upon the climatic conditions) by means of a pressure distributor at 275–350° F. at a uniform rate of 2 gals. (± 0.1 gal.) per sq. yd.

4. Immediately after applying the binder, and while it is still warm, spread a light sprinkling of ¾–¼-in. fine aggregate (broken stone, mine tailings or slag, of which 95–100 per cent passes ¾-in. screen, 25–75 per cent passes ½-in. screen and 0–5 per cent passes ¼-in. screen), and continue the rolling until smooth.

5. A second application of asphalt binder at a uniform rate of 0.4 gal. (± 0.1 gal.) per sq. yd. followed by a sprinkling of ¾–¼ in. fine aggregate, compacted by broom-dragging and rolling until smooth and free from depressions.

Bituminous Emulsions. When emulsions are employed, the procedure shall be as follows:

Penetration Method:

1. Level the foundation and prime with asphalt binder MC-1.

2. Construct the base-course by blading the aggregate over the surface in a uniformly thick, level layer; apply slow-setting asphalt emulsion with a pressure distributor; mix by blading once or twice; broom-drag and roll.

3. Construct the seal-coat by spreading the aggregate over the surface; broom-drag and roll; apply quick-setting asphalt emulsion with a pressure distributor; cover with stone-chips, broom-drag and roll. Apply a second coat of quick-setting emulsion and allow it to penetrate; cover with fine stone chips or coarse sand; broom-drag and roll.

Road-Mix Method:

1. Level the foundation and construct the base-course as in the foregoing.

2. Construct the seal-coat by spreading the aggregate in the center of the road; dampen with water until absorbed; apply slow-setting asphalt emulsion; mix with the aggregate by blading; distribute the mixture over the road in a uniform layer; level and roll. Apply a second coat of quick-setting emulsion; cover with fine stone chips or coarse sand; broom-drag and roll.

The following quantities have been recommended:

TABLE LXIV

QUANTITIES OF EMULSIONS USED IN BITUMINOUS MACADAM PAVEMENTS

(Quantities per square yard)

Compacted Finished Thickness (Inches)	Base-Course Coarse Stone		Appl. Slow-Setting Emulsified Asphalt† (Gals.)	Seal-Coat Keystone * ½"—¼" (Lbs.)	1st Appl. Quick-Setting Emulsified Asphalt (Gals.)	Stone Chips* ⅜ to 10-mesh (Lbs.)	2nd Appl. Quick-Setting Emulsified Asphalt (Gals.)	Sand or Stone Chips* ⅜ to 10-mesh (Lbs.)	Total Stone		Total Emulsified Asphalt (Gals.)
	Size	Lbs.							Lbs.	Cu. Yd.	
2½	2½"	200	0.6	25	0.5	12	0.4	8	245	.096	1.9
	to	to	to	to	to	to	to	to	to	to	to
	½"	210	0.8	30	0.7	18	0.6	12	260	.102	2.1
2	2"	140	0.5	20	0.5	12	0.4	8	190	.075	1.7
	to	to	to	to	to	to	to	to	to	to	to
	½"	160	0.7	25	0.7	18	0.6	12	210	.083	1.9
1½	1½"	110	0.4	15	0.5	12	0.4	8	145	.057	1.4
	to	to	to	to	to	to	to	to	to	to	to
	½"	125	0.5	20	0.6	18	0.6	12	170	.067	1.6
1¼	1"	100	0.4	10	0.4	10	0.3	8	125	.049	1.2
	to	to	to	to	to	to	to	to	to	to	to
	½"	115	0.5	20	0.5	15	0.5	12	150	059	1.5

(Assumed weight of aggregate 2550 lbs. per cubic yard.)

* Sizes indicated refer to Standard A.S.T.M. circular opening screens and 10-mesh sieves. Not more than 10 per cent. of the material shall be either coarser or finer than maximum and minimum sizes indicated.

† Certain types of aggregates may require slight increase in the maximum quantities of slow-setting emulsified asphalt shown above.

BITUMINOUS CONCRETE PAVEMENTS

These pavements differ from the foregoing in the fact that the mineral aggregate is heated and mixed with the bituminous binder *before* it is applied to the road. This overcomes the main difficulty of the penetration method and insures a *uniform* distribution of the bituminous material. The name "bituminous concrete" is given on account of its analogy to a Portland-cement concrete, in which the mineral aggregate has its voids filled with Portland cement in one case and with a bituminous cement in the other. It has also been termed "stone-filled sheet asphalt pavement," and may be regarded as a mixture of the composition used in the construction of the surface course of sheet asphalt pavements, with an aggregate uniformly distributed and embedded therein. The surface of the pavement is sometimes finished off with a seal-coat in the same manner as bituminous macadam. There are two types of bituminous concrete pavements, namely coarsely-graded and finely-graded. In the former the percentage of mineral particles larger than ¼ in. is greater than any other constituent of the mix, whereas in the latter type the percentage of mineral particles which pass a 10-mesh sieve is greater than any other constituent. This type of construction is rapidly superseding bituminous macadam, in spite of the fact that the latter is considerably less expensive. The modern tendency is to produce a very dense wearing course, characterized by a smaller proportion of voids than in bituminous macadam.

Warrenite and Bitulithic pavements are patented types of bituminous concrete pavements of the coarsely-graded type containing an asphaltic binder, and in which the fine surfacing mixture is incorporated during the process of construction. Amiesite is also a patented asphaltic concrete pavement which is constructed in two courses, the lower being composed of a coarsely-graded and the upper a finely-graded type. Both mixtures are laid cold in consequence of a special treatment of the asphalt with a liquefier and the use of lime. Another type of cold-laid pavement is produced by introducing the asphalt in two forms, one of which is mixed with the aggregate in a melted condition, and the other as powdered hard asphalt, which forms a dry protective coating, and serves to keep the particles from sticking together. When the pavement is rolled

under compression, the hard asphalt dissolves in the soft asphalt, forming a standard asphalt-cement. Such pavements have been introduced under the trade-names Colprovia, Sintex, etc. Still another variation consists in first coating the aggregate with the usual form of asphalt-cement, and when cool, the particles are coated with aluminium stearate which serves to keep them from adhering together, so that the material may be stored for a considerable length of time before it is transported and used.[87]

Topeka pavement is an unpatented asphaltic concrete pavement of the finely graded type in which the largest mineral particles pass a ½-in. screen. The original grading of the aggregate in this pavement was not as satisfactory as that now commonly used in what is known as the modified Topeka or stone-filled sheet-asphalt pavement. Other modifications are exploited under the names Bitoslag, Bitosan, Filbertine, etc.

Foundation. The most satisfactory foundation consists of a Portland-cement concrete, or a well compacted macadam; less satisfactory results being obtained from gravel or broken stone foundations. The same precautions should be followed in levelling and grading the macadam, broken stone or gravel foundation as described under the respective headings "Bituminous Surfacings" and "Bituminous Macadam." Macadam foundations should never be less than 6 in. thick, or Portland-cement concrete less than 4 in.

Base Course (commonly termed "black-base"). When asphaltic binders are employed, the base course shall be constructed as follows:[88]

1. The foundation (i.e., sub-base) must be dry and swept clean.

2. A mixture is prepared consisting of aggregates I-2 and P-1 heated to 225–350° F. with asphalt paving binder G, H, I, or J (depending upon the climatic conditions) heated to 250–350° F. in such proportions as to produce a composition of the following proportions by weight:

Passing 1½-in., retained on ¾-in. screen	20–30 per cent	50–65 per cent
Passing ¾-in. retained on ¼-in. screen	25–40 per cent	
Passing ¼-in. screen, retained on 10-mesh sieve	5–15 per cent ± 2.0 per cent	
Passing 10-mesh sieve	20–35 per cent ± 2.0 per cent	
Portion soluble in carbon disulfide	4– 7 per cent ± 0.5 per cent	

3. The foregoing mixture shall be delivered at 225–325° F.

and immediately spread with hot rakes in a layer which when compacted will have an average thickness of 3 in. Compress with a three-wheel power-driven roller weighing not less than 10 tons with a minimum weight of 200 lb. per inch width of rear wheel, at a rate not to exceed 200 sq. yds. per hour.

Surface Course—Coarsely-graded Aggregate Type.[89] This shall be constructed as follows:

1. The base course must be dry and swept clean.

2. A mixture is prepared consisting of aggregates I-1 and P-1 with filler S-1 heated to 225–350° F. with asphalt paving binder H, I, or J (depending upon the climatic conditions) heated to 250–350° F. in such proportions as to produce a composition of the following proportions by weight:

Passing 1¼-in., retained on ¾-in. screen	15–25 per cent
Passing ¾-in. retained on ½-in. screen	20–25 per cent
Passing ½-in. screen, retained on 10-mesh sieve	5–15 per cent ± 2.0 per cent
Passing 10-mesh retained on 200-mesh sieve	25–35 per cent ± 2.0 per cent
Passing 200-mesh sieve	4– 6 per cent ± 1.5 per cent
Portion soluble in carbon disulfide	5– 8 per cent ± 0.5 per cent

3. The foregoing mixture shall be delivered at 225–325° F. and immediately spread with hot rakes in a layer which when compacted will have a thickness of 2 in. (± ¼ in.). Compress with a three-wheel power-driven roller weighing not less than 10 tons with a minimum weight of 200 lb. per inch width of rear wheel, at a rate not to exceed 150 sq. yds. per hour. After compression the surface course shall at no point have a density of less than 95 per cent of the calculated maximum density of a voidless mixture composed of the same materials in like proportions.

Surface Course—Finely-graded Aggregate Type.[90] This is sometimes termed "stone-filled sheet-asphalt," and is constructed as follows:

1. The base course must be dry and swept clean.

2. A mixture is prepared consisting of aggregates L-2 and Q-2 with filler S-1 heated to 225–350° F. with asphalt paving binder F, G, H, I or J (depending upon the climatic conditions) heated to 250–350° F. in such proportions as to produce a composition of the following proportions by weight:

Passing ⅜-in. screen, retained on 10-mesh sieve	25–35 per cent
Passing 10-mesh, retained on 40-mesh sieve	7–30 per cent
Passing 40-mesh, retained on 80-mesh sieve	11–40 per cent
Passing 80-mesh, retained on 200-mesh sieve	10–30 per cent ± 2.0 per cent
Passing 200-mesh sieve	7–12 per cent ± 1.5 per cent
Portion soluble in carbon disulfide	7–9.5 per cent ± 0.5 per cent

3. The foregoing mixture shall be delivered at 225–325° F.

and immediately spread with hot rakes in a layer which when compacted will have a thickness of 2 in. (± ¼ in.). Compress with a three-wheel power-driven roller weighing not less than 10 tons, with a minimum weight of 200 lb. per inch width of rear wheel, at a rate not to exceed 150 sq. yds. per hour. After compression the surface course shall at no point have a density less than 94 per cent of the calculated maximum density possible to obtain a voidless mixture composed of the same materials in like proportions.

Asphalt Emulsions.[91] When emulsions are employed, the procedure shall be as follows:

The aggregate is spread on the previously primed foundation and then wet by sprinkling with water. Apply a slow-setting type of emulsion with a pressure distributor at the rate of 2 to 2½ gal. per sq. yd. for each 2-in. compacted thickness, followed immediately with a harrow, and finally bladed 3 to 5 times to insure uniform mixing. As soon as the mixture sets, it is rolled.

The mixture may alternately be prepared in a batch-mixer alongside of the road as follows: The aggregate is first moistened with water at the rate of 2½ gal. per 750 lb. aggregate. The emulsion is then added and allowed to mix for about half a minute, whereupon it is spread on the road and raked in place. When set, it is rolled with an 8-ton tandem roller.

It has been suggested that melted asphaltic road binders will form a better bond with aggregates, if the latter are first coated with a bituminous emulsion.[92]

SHEET-ASPHALT PAVEMENTS[93]

A sheet-asphalt pavement is one prepared by the mixing method, composed of an intermediate or binder course of bituminous concrete, and a wearing course composed of asphaltic cement and sand of *predetermined grading,* with or without the addition of mineral filler (dust). The wearing course is often called "sheet-asphalt topping" or "surface mixture" and since it is compacted under pressure it is also referred to as "compressed asphalt" (French: *asphalte comprimé;* German: *Stampfasphalt*) in distinction to mastic asphalt, which is poured into place. The binder course is asphaltic concrete of the finely-graded type, and is used as an intermediate course between the foundation course and the wearing course.

Stone-filled sheet-asphalt pavements consist of a finely-graded aggregate asphaltic-concrete in which the aggregate passing a 10-

mesh sieve conforms to the usual grading requirements of the sheet-asphalt surface mixture. A sand-asphalt pavement is one in which the asphalt is mixed with local sand which does not comply with the accepted grading requirements for sheet-asphalt, and is therefore inferior to sheet-asphalt. A rock-asphalt pavement is one in which the wearing course is constructed of natural rock asphalt, which has been crushed and sometimes fluxed with additional asphalt or flux oil. Asphaltic sandstone, such as Kentucky rock-asphalt is laid cold, whereas asphaltic limestone, either as such, or combined with asphaltic sandstone, is usually worked up as a hot mixture, and laid in the same manner as sheet-asphalt pavements.

Coal-tar products are not used in sheet-asphalt pavements.

Foundation. The foundation may consist of a brick or a block pavement, and sometimes old macadam. The most satisfactory foundation is composed of Portland-cement concrete 4 to 9 in. thick, depending upon the severity of traffic. The concrete [94] should consist of Portland cement, gravel or broken stone, and sand in proportions ranging from 1 : 2 : 5 to 1 : 3 : 6 (preferably the latter), depending upon the nature of the aggregate available. The surface of the hydraulic concrete should be levelled carefully and finished smooth, since any irregularities will be transmitted to the wearing course, giving it an uneven surface. To promote better adhesion of the asphaltic binder or intermediate course, it has been suggested that an asphalt emulsion either be applied as a coating over the concrete foundation, or else be mixed with the constituents of the concrete in the course of its preparation.[95]

Binder and Surface Courses.[96] These shall be constructed in the following manner:

1. The foundation (i.e., sub-base) must be dry and swept clean.

2. The binder course is prepared by mixing aggregates I-3 and O (or as alternates P-1 or P-2) heated to 225–350° F. with asphalt paving binder F, G, H, I or J (depending upon the climatic conditions) heated to 250–350° F. in such proportions as to produce a composition of the following proportions by weight:

Retained on ½-in. screen	52–72 per cent (± 2.0 per cent)
Passing ½-in. screen, retained on 10-mesh sieve	8–20 per cent (± 2.0 per cent)
Passing 10-mesh sieve	15–35 per cent (± 2.0 per cent)
Portion soluble in carbon disulfide	4– 6 per cent (± 0.5 per cent)

3. The foregoing mixture shall be delivered at 225–325° F. and immediately spread with hot rakes in a layer which when compacted will have a thickness of 1½ in. (± ½ in.). Compress with a three-wheel power-driven roller weighing not less than 10 tons with a minimum weight of 200 lb. per inch width of rear wheel, at a rate not to exceed 300 sq. yd. per hour.

4. The surface course is prepared by mixing aggregates I-3 and Q-2 and mineral filler S-1 heated to 275–400° F. with asphalt paving binder F, G, H, I or J (depending upon the climatic conditions) heated to 250–350° F. in such proportions as to produce a composition of the following proportions by weight:

Passing 10-mesh, retained on 40-mesh sieve.....	10–40 per cent
Passing 40-mesh, retained on 80-mesh sieve.....	20–45 per cent
Passing 80-mesh, retained on 200-mesh sieve.....	12–32 per cent (± 2.0 per cent)
Passing 200-mesh sieve.....	10–20 per cent (± 1.5 per cent)
Portion soluble in carbon disulfide	9.5–12 per cent (± 0.5 per cent)

5. The foregoing mixture shall be delivered at 250–375° F. and immediately spread with hot rakes in a layer which when compacted will have a thickness of 1½ in. (± ¼ in.). Compress with a three-wheel power-driven roller weighing not less than 10 tons with a minimum weight of 200 lb. per inch width of rear wheel, at a rate not to exceed 150 sq. yds. per hour. After compression the surface course shall at no point have a density less than 94 per cent of the calculated maximum density possible to obtain a voidless mixture composed of the same materials in like proportions.

General Considerations.

A properly balanced mineral aggregate should contain sufficiently fine material "passing 200-mesh," to make the compressed pavement dense, tough, slightly susceptible to temperature changes, unaffected by water, and not liable to displacement in service. Too much material "passing 200-mesh" will cause the pavement to "ball up" and prevent it from spreading easily while in a heated condition; it is also apt to make the finished pavement "mushy" and liable to indentations; and it will consume an excess of asphaltic cement, at a correspondingly increased expense. The fine material "passing 200-mesh" is usually added in the form of "filler," or "dust" the quantity of which will depend largely upon whether or not the asphaltic cement itself contains mineral matter. The quantity of filler to be added to the sand should be regulated so that the surface mixture will contain not less than 10 per cent "passing 200-mesh"

when the pavement is to be subjected to moderate traffic conditions, nor more than 20 per cent, when intended to withstand heavy traffic.

The aggregate must contain sufficient particles "passing 80—and retained on a 200-mesh sieve" to overcome the tendency of the filler to "ball up" the surface mixture while it is being spread on the pavement, enabling the mixture to spread out easily and form a dense surface under compression, capable of resisting the action of water.

The particles "passing 10—and retained on a 40-mesh sieve" form the "backbone" of the pavement, as it were, preventing the mixture from becoming displaced in service, and forming a less slippery wearing surface.

It is unusual to find one sand fulfilling all of the foregoing requirements. Generally two or more sands must be blended together, and a suitable proportion of filler added, to produce an aggregate of the optimum characteristics. Richardson has patented a mixture containing at least 10 per cent mineral particles passing 200-, 100- and 80-mesh sieves respectively, and not less than 10½ per cent pure asphalt.[97] For an elaborate series of mathematical calculations, including formulas, to determine the proper grading of mineral aggregates so as to produce a mixture of the optimum characteristics, the reader is referred to the publications of R. Wilhelmi.[98]

Asphaltic cement should be present in just sufficient quantity to completely fill the voids in the aggregate after compression. Too little will form a pavement lacking strength, and liable to crack upon being subjected to changes in temperature. Too large a proportion will make the surface mixture "mushy" so that the particles of sand are apt to become displaced under traffic. Modern practice calls for 9 to 12½ per cent of asphaltic cement, depending upon the character of the aggregate and the conditions under which the pavement will be subjected. It has been shown [99] that the mixing operation tends to increase the fusing-point and hardness, also to decrease the ductility of the asphalt, similar to the changes brought about by blowing the product.

With different characteristics of the aggregate and filler, the proportions of the constituents will vary to a considerable extent, nevertheless the following percentages by weight represent a fair average: sand 75 per cent, filler 15 per cent, asphaltic cement 10

per cent. When the pavement is compressed on the street, it is seldom free from voids, which range from 1.5 to 5 per cent by volume (Test 43). The most suitable proportions of any given set of constituents may best be ascertained by running a series of "stability tests" (Test 47) on various admixtures. For heavy traffic conditions the stability value should exceed 2000 lb., and the minimum safe limit for light traffic is 1200 lb.

Many native asphalts carrying mineral matter are adapted for constructing the surface course of sheet-asphalt pavements, but in these the mineral matter associated with the asphalt should substitute a portion of the aggregate to be added. Thus in the case of the Trinidad asphaltic cement, containing naturally a percentage of clay and silica, 20 per cent less filler must be added than with a Bermudez asphaltic cement almost free from associated mineral constituents. In Europe, the Sicilian asphalt deposits are most extensively used for this purpose, and particularly that obtained from Ragusa, which contains 8 to 9 per cent soluble in carbon disulfide, averaging 8.4 per cent, associated with a pure limestone base. The soluble matter is quite soft and very stable on heating. The rock as mined contains 3.05 per cent moisture and 0.67 to 0.74 per cent volatile at 325° F. in five hours (Test 16). Limmer and Val de Travers asphalts show a higher percentage of volatile matter. The original Val de Travers asphalt enjoyed a good reputation for pavements, but now an inferior quality is mined.[100] European pavements carry 10½ to 11 per cent of asphalt, having a penetration at 77° F. of 40 to 60, although there is now a tendency towards harder mixtures, having a penetration between 30 and 40. A mixture composed of the proper ingredients, suitable for shipment and storage, and adapted to being converted into A.C. on the job by heating, consists of powdered hard asphalt and the mineral aggregate with filler in conjunction with sufficient asphaltic flux, mixed with the former in the cold and compressed in the form of cakes.[101]

The deterioration of sheet asphalt pavements is due to:

(1) Defective construction, including improper proportioning of the ingredients.

(2) The actual wear and tear of traffic.

(3) Aging due to exposure to the elements.

Worn-out pavements may be repaired by softening and remov-

ing the surface by means of an oil-heater; [102] or by a device involving the use of molten metals (e.g., lead); [103] or by means of live steam; [104] followed by resurfacing with some of the freshly prepared mixture. The use of the surface-course ripped from old pavements, fluxed with additional A.C. has been proposed for use in resurfacing.[105]

BITUMINOUS JOINT-FILLERS

A bituminous filler is the term applied to a bituminous composition which is introduced into the joints of wood-block, asphalt-block, granite-block and brick pavements, also for introducing into the joints between sections of Portland-cement concrete pavements, by melting and pouring. There are three general types of fillers, viz.: a poured filler consisting of a prepared bituminous material (without the addition of extraneous mineral matter) intended to be melted and poured into the joints; a grout filler consisting of a mechanical mixture of bituminous matter and fine sand, intended to be poured while hot over the pavement and broomed or squeegeed into the joints; also a prepared joint-filler which consists of a premolded composition, which will be described under the heading "Bituminous Expansion Joints." The filler must adhere to the blocks, bricks or concrete so as not to loosen or chip in cold weather under the impact of traffic or when subjected to the strains brought about by contraction or settling of the pavement, and it must also be sufficiently resistant to high temperatures so as not to exude from the surface or run out of the joints.

Asphaltic binders H, I and J used in the form of a grout by mixing with about equal parts of hot sand (Aggregates O and R), are intended for filling stone-block and brick pavements. Asphaltic binder H used in the form of a grout is used for filling asphalt-block and brick pavements.[106] Asphaltic binder N is used without admixture of sand for filling asphalt-block and brick pavements, and also the joints between sections of concrete pavements. Pitch fillers W, X and Y are used as such, or in the form of a grout for filling stone-block, brick, concrete or wood-block pavements.

Filling the Joints. With brick- and stone-block pavements, the courses should be laid with joints ⅜ to ½ in. wide. When a pitch filler is to be used, the joints are half filled with hot ⅛ to ⅜ in.

gravel (containing not over 25 per cent of the ⅜-in. size). Before the gravel cools, the joints should be filled half way to the top with filler, then to within ½ in. of the surface with hot gravel, and lastly poured full with filler. In performing the work, the pitch should be heated from 250 to 325° F., but never exceeding the latter. When asphaltic filler is used, sand or gravel are unnecessary. The filler is heated from 300 to 450° F. and poured into the joints until completely filled. A top dressing of sand should be spread over the filler while hot to form a wearing surface.

The joints of asphalt-block and wood-block pavements are formed as closely as possible, and the melted filler poured over the entire pavement and worked into the joints until flush by scraping with a wooden or rubber squeegee, or a broom, whereupon the entire pavement is sprinkled with fine sand or gravel. Standard block-pavements require 1¼ to 1½ gal. filler to the square yard.

Portland-cement concrete pavements are laid in sections with ½-in. joints which are subsequently poured full of the filler, which is likewise used to fill any cracks which may subsequently develop in the pavement through settling.

ASPHALT-BLOCK PAVEMENTS

These constitute pavements in which the wearing course is constructed of premolded blocks composed of a dense, finely-graded aggregate asphaltic-concrete, which has been subjected to heavy compression during the process of molding. These blocks are laid in regular courses, as in the case of brick and stone-block pavements. The manufacture of asphalt blocks was begun in San Francisco in 1869, but it was not until 1880 that the perfection of a powerful mechanical press and greater skill in proportioning the ingredients placed them on a firm basis.

The blocks are made of coarse aggregate, dust filler and asphaltic binder. The latter shall consist of binder E of either 10–15 or 15–25 penetration, depending upon traffic and climatic conditions. To produce a good block, the binder should possess sufficient cohesiveness, tensile strength and stability (i.e., non-hardening with age), and must be non-friable at low temperatures and show the minimum softening at the highest atmospheric temperatures.

The coarse aggregate should consist of crushed hard rock or copper conglomerate (a by-product from copper mines). The crushed rock should be clean, hard, unweathered rock which should show a coefficient of wear of more than 5, a hardness not less than 16 and a toughness of not less than 12. The coarse aggregate should be as nearly cubical as possible and not less than 97 per cent shall pass a screen having ¼-in. circular openings. It is advisable that there should be present at least 20 per cent of filler, preferably ground limestone, of which at least 90 per cent will pass a 30-mesh sieve and 50 per cent a 200-mesh sieve. From 6 to 9 per cent by weight of asphaltic binder shall be used. The heated mixture is compressed in molds at 274 to 300° F. under a pressure of 4 to 5 tons per sq. in. The blocks should show a specific gravity at 77° F. of not less than 2.45 if trap-rock filler is used, or not less than 2.35 if the filler is composed of limestone.

Asphalt blocks are now made in the following sizes:

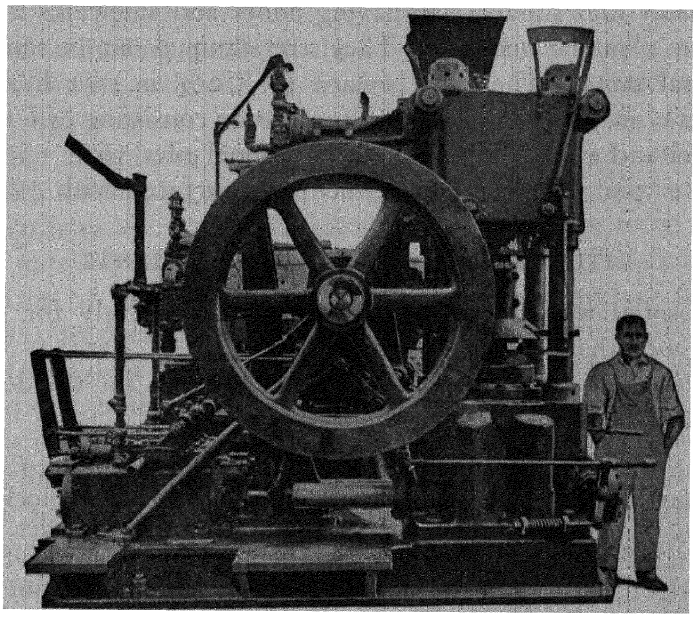
Length, Inches	Width, Inches	Depth, Inches	Weight, Pounds
12	5	2	11
12	5	2½	13½
12	5	3	16
8	4	1½	3½

The first listed block is generally used in pavements and the deep sizes are intended for specially severe conditions. The last-named block, known as "eight-four" is designed to meet a minimum price consistent with durability under moderate conditions of wear. Fig. 141 shows a high-speed press capable of turning out 40 blocks per minute and is capable of subjecting them to a pressure of 300 tons per block. The blocks are cooled under water on a conveyor belt. The composition of the blocks should conform to the following percentages by weight:[107]

Retained by ½-inch screen	Max.	3 per cent
Passing ½-inch screen and retained on 20-mesh sieve		35–60 per cent
Passing 20-mesh sieve and retained on 100-mesh sieve		15–30 per cent
Passing 100-mesh sieve (including all fines)		20–35 per cent
Passing 200-mesh sieve	Min.	15 per cent
Portion soluble in carbon disulfide		6½–10 per cent

Foundation. Asphalt blocks may be laid on a foundation consisting of gravel, macadam, or preferably concrete. The foundation course should be covered with a "cushion" or "bedding course," composed either of sand 1 in. thick, or cement mortar ½ in. thick, and the blocks embedded in the latter before it commences to set.

Laying the Blocks. Blocks are laid closely together, resting on the 12 by 6-in. surfaces extending lengthwise across the street, the



Courtesy Hastings Paving Co.

FIG. 141.—High-speed Press for Making Asphalt Paving Blocks.

joints being broken 4 in. They should be wedged together as firmly as possible to close the joints between them, and rammed into place. After being laid, the blocks are given a light coat of sharp, fine sand, well broomed into the joints, and the pavement opened to traffic in three to four days.

Asphalt blocks have improved greatly in the last few years and are now capable of withstanding severe traffic conditions without fracturing. They are slightly more expensive than a sheet asphalt pavement, but permit repairs being effected more readily, and insure a more uniform composition also a greater freedom from structural faults. When properly made, the blocks will weld together after a

time, upon being subjected to traffic, so that the joints between them will become almost invisible. Sometimes they are anchored in place with metal rods to prevent the blocks shifting. If the binder is too hard and brittle, instead of the blocks welding, the edges will chip and spall. On the other hand, if the binder is too soft or fusible, the blocks will lose their shape, becoming distorted, even before they are laid in place.

Asphalt tiles suitable for paving floors and sidewalks are prepared in a similar manner. They are stamped out in square or hexagonal units. The large square ($8 \times 2\frac{1}{4}$ in.) or hexagonal ($10 \times 2\frac{1}{4}$ in.) tiles are laid on a foundation consisting of 6 to 8 in. of gravel and sand, and the small hexagonal tiles (6×1 in.) are laid on a 3-in. foundation, in either case surfaced with $\frac{1}{2}$ in. of mortar.

BITUMINOUS WOOD-BLOCK PAVEMENTS

Creosoted wood-blocks are being used extensively for paving roads, footpaths, floors of buildings, etc.

Methods of Impregnation. The specifications for wooden paving blocks,[108] provide that:

"The wood, which shall be treated, shall be Southern yellow pine, Douglas fir, tamarack, Norway pine, hemlock, or block gum. Only one kind of wood shall be used in any one contract. The blocks shall be sound and shall be well manufactured, square-butted, square edged, free from unsound, loose or hollow knots, knot holes, worm holes and other defects such as shakes, checks, etc., that would be detrimental to the blocks.

The number of annual rings in the 1 in. which begins 2 in. from the pith of the block shall not be less than six, measured radially; provided, however, that blocks containing between five and six rings in this inch shall be accepted if they contain $33\frac{1}{3}$ per cent or more summerwood. In case the block does not contain the pith, the 1 in. to be used shall begin 1 in. away from the ring which is nearest to the heart of the block. The blocks in each charge shall contain an average of at least 70 per cent of heartwood. No one block shall be accepted that contains less than 50 per cent of heartwood."

The size of the blocks ranges as follows:

Depth between 3 and 4 in., depending upon the severity of the traffic to which they are to be subjected, usually 3 in. for light traffic, $3\frac{1}{2}$ for medium and 4 in. for heavy traffic conditions.

Width should be *uniform* for any particular pavement, but may vary between 3 and 4 in. The best practice provides that there should be a difference of not less than ¼ in. between the width and the depth, which under no circumstances should be made equal.

The length will vary in any particular lot between 5 and 10 in., averaging about 8 in. Blocks 3 in. deep should not exceed 8 in length, blocks 3½ in. deep should not exceed 9, and blocks 4 in. deep should not exceed 10.

After removing the bark, the wood is cut into planks equalling in thickness the width of the finished block. The planks are then run through a planer, trimming them all to *exactly* the same size (insuring a uniform width of the finished blocks), and finally through a set of gang-saws, which cuts them into the finished blocks.

Formerly, the wood was seasoned out of doors from four to twelve months, depending upon the kind of wood, its dimensions, the season of the year and the locality. At the present time this process is accelerated by steaming the blocks for two to four hours in steel cylinders varying in size from 6 ft. in diameter by 42 ft. long, to 9 ft. in diameter by 172 ft. long.[109] In this treatment, live steam is introduced at not exceeding 20 lb. pressure per square inch, raising the temperature between 220 and 240° F. for not less than two nor more than four hours. After the steaming, the cylinder is subjected to a vacuum of not less than 22 in. maintained for at least one hour, to withdraw the moisture and resinous matters. This is continued until the blocks are thoroughly dry, whereupon the creosote oil heated to 180–220° F., or a mixture containing 80 per cent zinc chloride and 20 per cent creosote is introduced while the vacuum is still on, care being taken that no air is admitted. The pressure is applied gradually, not to exceed 50 lb. at the end of the first hour, or 100 lb. at the end of the second hour, and then maintained at not less than 100 nor more than 150 lb. until the proper impregnation has been attained. The oil is pumped from the cylinder and the blocks allowed to drain for about a half hour at a temperature of 200° F. under a vacuum of at least 20 in. for not less than 30 minutes. The quantity of preservative introduced should range between 6 and 12 lb. per cubic foot with blocks used for floors, and at least 16 lb. per cubic foot with blocks used for paving roads. The function of the preservative is twofold, namely:

(1) To prevent the wood from decaying, due to the ravages of fungi and molds.

(2) To waterproof the blocks, preventing them from warping or swelling.

Ten pounds of the preservative per cubic foot are sufficient to preserve the blocks from decay, although a larger quantity must be used to secure the required waterproof properties. To completely waterproof the blocks, 25 lb. would be required, which is more than ordinarily used in practice.

Creosote Preservatives. The so-called "creosote" preservatives include the following products:

(1) Distillates from gas-works coal tar or coke-oven coal tar, known commercially as "distillate oil."

(2) Mixtures of the foregoing "distillate oil" with not exceeding 35 per cent of a gas-works coal tar or coke-oven coal tar, containing preferably 5, but not exceeding 25 per cent free carbon. Refined or filtered tars are recommended for this purpose.

The following definitions have been proposed [110] for the aforesaid products:

Creosote (Creosote Oil, Creosote Distillate). As used in wood preservation, a distillate of coal tar produced by high-temperature carbonization of bituminous coal; it consists principally of liquid and solid aromatic hydrocarbons, and contains appreciable quantities of tar acids and tar bases; it is heavier than water; and has a continuous boiling range of at least 125° C. beginning at about 200° C.

Creosote-Coal Tar Solution. Solution of coal tar in creosote in various proportions. Usually contains 20 to 40 per cent of coal tar.

Table LXV contains the specifications of various grades of creosote oil as well as mixtures with coal tar and water-gas tar,[111] in which B, D, E and F represent distillates from coal-gas tar or coke-oven tar; H a distillate from coal tar or coke-oven tar; I a distillate from water-gas tar; A a mixture of 65 per cent distillate, and G a mixture of 80 per cent distillate from coal-gas tar or coke-oven tar, and the balance refined or filtered coal-gas tar or coke-oven tar; C refined water-gas tar; and J a mixture of 60 per cent distillate from water-gas tar, and the balance refined or filtered water-gas tar.

TABLE LXV

Specifications for Creosote Oil and Mixtures with Coal-tar or Water-gas Tar

Test No.	Description of Test	For Paving Blocks and Flooring			For Ties and Structural Timber				For Brush and Spray Treatment	For Use with Zinc Chloride	
		Coal-tar Paving Oil*	Coal-tar Distillate Oil*	Refined Water-gas Tar	Creosote Oil Grade 1*	Creosote Oil Grade 2*	Creosote Oil Grade 3*	Creosote Coal-tar Solution*	Coal-tar or Coke-oven Tar Distillate*	Water-gas Tar Distillate	Water-gas Tar Solution
		A	B	C	D	E	F	G	H	I	J
7a	Specific gravity at 38°/15.5° C.....Min.	1.07–1.14	1.06	1.11–1.14	1 03	1.03	1 03	1 05–1.12	1.06	1 02	1.03–1.07
16b	Distillate (water-free material):										
	Start to 210° C. (per cent by weight).....Max.	5	5	5	5	8	10	5	1	5	8
	Start to 235° C. (per cent by weight).... Max.	25	15	15	25	35	40	25	10	25	20
	Start to 315° C. (per cent by weight)... .Max.	.....	.....	40							
	Start to 355° C. (per cent by weight).....Min.	.....	.....	25	.....	.....	.....	.....	65	70	60
7a	Total distillate to 355° C., sp. gr. at 38° C./15.5° C.....Min.	.....	.....	0.99–1.02							
	Provided the residue at 355° C. exceeds the stipulated per cent:.....	35	10	.....	5	5	5	26	10	5	25
8d	Then float test of residue at 70° C., shall be, secs.....Max.	80	50	.....	50	50	50	50	50	50	50
19	Coke residue (i.e., fixed carbon), per cent...Max.	10	2	.....	2	2	2	6	2	2	10
24	Insoluble in benzol, per cent.....Max.	3	0.5	2	0 5	0.5	0.5	2	0.5	0.5	2
25	Water, per cent.....Max.	3	3	3	3	3	3	3	1	3	3

* The following tests will determine the creosote to be of a coal-tar origin:

(*a*) The fraction between 210 and 235° C. is either a solid or contains solids when cooled to 25° C.

(*b*) All fractions up to 315° C. shall contain at least 1 per cent of tar acids (Test 31).

(*c*) The fraction between 235 and 315° C. shall have a specific gravity at 38° C./15.5° C. of not less than 1.025.

(*d*) The fraction between 315 and 355° C. shall have a specific gravity at 38° C./15.5° C. of not less than 1.085.

Mixtures of distillate oil with a certain proportion of refined tar are supplanting the use of the oil alone, for the reasons that:

(1) They are less expensive.
(2) They waterproof the blocks more efficiently.
(3) They volatilize less readily.

The presence of too much tar in the creosote mixture interferes with the penetration, due to the fact that the tar has a greater viscosity than the distillate oil at high temperatures. An excess of free carbon in the tar will clog the pores of the block and similarly interfere with the penetration.

Various other substances have been proposed for this purpose, including asphalt;[112] Edeleanu extract;[113] mixtures of creosote oil with fatty-acid pitch;[114] etc.

Foundation. This will vary in thickness depending upon the traffic, consisting of a Portland-cement concrete (Aggregate P_2) 5 to 9 in. thick, usually averaging 6 in. A 1 : 2½ : 5 concrete with a smooth-trowelled finish is recommended.[115]

Cushion Layer. The best practice consists in embedding the blocks in a "cushion" of melted coal-tar pitch mixed with sand (Aggregate O) (termed "paint-coat"), or a bituminous mastic composition, or a mortar of Portland cement. These will be considered:

(*a*) Coal-tar pitch Paint-coat Construction: The same character of coal-tar pitch as used for filling the joints is melted and poured over the foundation in a uniform layer averaging ⅛ in. thick.

(*b*) Bituminous-mastic Construction: A mastic is prepared consisting of 10 per cent of either coal tar or asphaltic oil and 90 per cent by weight of clean, dry sand, slag, limestone screenings, etc.,[116] which is melted and applied to the foundation in a layer averaging ¾ in. thick.

(*c*) Mortar-bed Construction:[117] This alternative consists in spreading over the foundation a 1 : 3 Portland-cement mortar in a layer averaging ¾ in. thick, and the blocks immediately rammed into place, so as to form a level surface free from depressions.

The English practice, which now seems to be gaining favor in this country, consists in constructing the foundation course as smooth and level as possible, laying the blocks in place, and pouring hot tar between the crevices. The tar will first work its way underneath the blocks and cement them to the foundation, and the pouring is

continued until the joints are filled half way to the surface. The joints are then filled with a grout of Portland cement. This procedure prevents "bleeding."

The blocks may be laid in rows either perpendicularly to the curb, or at angles varying from 45° to 67½°, the latter having the advantage of conforming to the expansion and contraction of the pavement without the necessity of providing transverse expansion joints. Where the blocks are laid at right angles to the curb, ½ in. transverse expansion joints should be introduced every 50 or 60 ft. In all cases, an expansion joint ¾ in. wide, should be provided between the blocks and the curb, composed either of a preformed bituminous strip, or a melted bituminous mixture having a penetration of 30–50 at 77° F. (Test 9*b*). The blocks must be laid as closely together as possible, with joints not exceeding ⅛ in., and levelled with a roller weighing not less than 4 tons.

Filling the Joints. Either a coal-tar pitch or asphaltic filler[118] may be used. Coal-tar pitch fillers shall be derived from the distillation of coal tar and shall conform with the requirements of paving binders N and Y.[119]

The bituminous filler is swept over the surface of the blocks while in a melted condition, and has the advantage of augmenting the waterproof properties of the blocks, at the same time providing for expansion and contraction. The filler is covered with sand or stone chips, or with a cement grout to obviate any tendency of tracking.

General Considerations. A wood-block pavement when properly constructed is extremely durable even under heavy traffic; it may readily be repaired and is less noisy than any other bituminous pavement. The disadvantages are its slipperiness in damp or snowy weather, and the fact that under certain conditions it is apt to "bleed." The latter manifests itself by the soft bituminous matter exuding in summer, and being readily tracked about.

Bleeding is caused by introducing too large a proportion of the creosote mixture into the blocks, and is likely to occur when more than 16 lb. are used per cubic foot. It will also occur when the pavement is not provided with expansion joints, in which event the compression brought about by expansion will squeeze some of the creosote from the pores of the blocks.

ASPHALT MASTIC FOOT-PAVEMENTS AND FLOORS

In the United States asphalt mastic is restricted to the construction of foot-pavements and floors, for waterproofing railway bridges, also for lining reservoirs and tanks. In composition, it is similar to the asphaltic concrete, but it contains a larger percentage of asphaltic-cement, so that it may be laid with greater facility. It is differentiated from bituminous concrete, sheet-asphalt pavements, etc., by the fact that the asphalt mastic on being heated forms a thick and slowly flowing mass which may be *poured* into place. The mixture on being allowed to cool partially, is compressed by hand-trowelling to a smooth surface. Asphaltic concrete and sheet asphalt pavements on the other hand, do not melt when heated, but form an adherent, powdery mass, which upon being shovelled into place and raked smooth, require compression underneath heavy rollers, properly to compact them. Asphalt mastic is known as "asphalte coulé" in France and "Guszasphalt" in Germany.[120]

Acid-resisting mastics[121] are used for the following purposes:

(*a*) Floors in battery rooms, sugar refineries, acid and chemical works, artificial silk factories, breweries, etc.

(*b*) For lining tanks and pits, usually constructed of wood (e.g., oak) in metallurgical plants, pickling and galvanizing processes, vinegar storage, brewing vats, etc., in which cases the acid-proof mastic is applied in 2 layers, each ⅜ in. thick.

(*c*) Lining ducts and flumes intended for conveying corrosive liquids.

Asphalts Used. Asphalt mastic work constituted the earliest type of asphalt pavement, and few changes have been made in its mode of application since the inception of the industry. The first products used in asphalt mastic construction were the native "rock asphalts," including Val de Travers, Limmer, Seyssel, and later on Ragusa which are known in Europe under the generic term "asphalte," as was originally used in France. The refined rock asphalt (previously heated to expel the moisture and volatile oils) was first "cooked" over an open fire in a suitable melting tank provided with a stirrer, and combined with a purer native asphalt, for example, Trinidad (of which about 8 or 10 per cent was used), Bermudez, or with a residual asphalt, so the finished batch carried 12 to 18 per cent of pure asphalt, the balance being composed of

finely-divided mineral matter (calcium carbonate, silica, iron and aluminium oxides, etc.). Sometimes asbestos fibers are incorporated.[122] The admixture of shredded rubberized fabric (e.g., discarded automobile tires) has also been suggested.[123]

For convenience in shipping and handling, the mass is run into suitable molds and thus cast into flat cakes weighing 50 to 75 lb. each, customarily stamped with the name of the asphalt used. These cakes were known as "rock-asphalt mastic." To-day they are prepared from Trinidad asphalt, mixtures of asphaltites and flux, blown petroleum asphalts and residual asphalts, together with calcium carbonate or finely powdered silica, all the ingredients being combined at once in a portable rotary mixer. The native rock asphalts first mentioned are all still being used abroad.

It follows, therefore, that asphalt mastic includes (1) a mixture containing asphaltic material as a binder and graded mineral matter as an aggregate; or, (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature (usually 400° F.) may be poured into place, but which requires trowelling to form it into a compact mass. It is used for surfacing and waterproofing floors, bridges, subways, tanks, reservoirs, etc. The so-called "acid-proof mastics" are used extensively in chemical plants, metal pickling, plating, acid tanks, storage battery rooms, chemical laboratories, copper, gold, lead and other refineries.

Methods of Preparation. Asphalt mastic is prepared by combining together:[124]

(*a*) Asphalt mastic cake	48 per cent by weight
(*b*) Asphalt cement	5 per cent by weight
(*c*) Mineral aggregate	47 per cent by weight
Total	100 per cent by weight

(*a*) Asphalt mastic cake (sometimes termed "asphalt mastic block") consists of a mixture containing asphaltic material as a binder and an aggregate composed chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" (adapted for use in preparing asphalt mastic), conforming to the following:

Weight per cake	Maximum, 75 lb.*
Soluble in benzol	14–18 per cent by weight.
Insoluble on benzol	82–86 per cent by weight

*For acid-proof mastic: Maximum 80 lb.

The cake is composed of asphalt cement similar to (*b*), in combination with fine mineral filler, 40 to 60 per cent, and the balance sharp-grained sand. The filler may consist of limestone dust, Portland cement, dolomite dust, or similar material corresponding to aggregate Type S_1 or S_2. In the case of acid-proof mastic, it shall consist of a siliceous filler,[125] e.g., powdered pumice stone;[126] or a mixture of aluminium silicate and barium sulfate.[127]

(*b*) Asphalt cement, sometimes termed "soft flux," shall conform to asphalt binder, Type F (25–30 penetration). This is sometimes specified to have in addition a fusing-point of 140–160° F. and a penetration at 115° F. or 140 maximum. A so-called "hard flux" is sometimes desired, consisting of either a refined or fluxed Trinidad asphalt or a petroleum asphalt of the following characteristics:

(Test 9*b*)	Penetration at 115° F.	Max. 20
	Penetration at 77° F.	Max. 10
(Test 15*b*)	Fusing-point (R. and B. method)	180–210° F.
(Test 16)	Volatile at 325° F., 5 hours	Max. 1.5 per cent
(Test 17*b*)	Flash-point (open cup)	Min. 392° F.

(*c*) Mineral aggregate shall be either sand or gravel. In the case of acid-proof mastic there shall be no limestone present. It shall be clean, hard-grained and free from clay, silt, organic or other foreign matter, and shall be properly graded from coarse to fine particles so as to produce a mixture of the greatest density and stability, without interfering with the proper mixing or working of the materials. It shall have the following granularmetric composition:

Passing 200-mesh sieve	None	
Passing 50-mesh sieve and retained on 200-mesh sieve	Max.	25 per cent
Passing 10-mesh sieve and retained on 50-mesh sieve	Min.	25 per cent
Passing 4-mesh sieve and retained on 10-mesh sieve	Min.	50 per cent
Retained on 4-mesh sieve	Max.	10 per cent

(*d*) Completed mastic mixture, upon extraction with benzol shall test as follows:

Recovered Asphalt: This shall conform with the specifications for asphalt binder Type F.

Recovered Mineral Aggregate: This shall test as follows:

Passing 200-mesh sieve	Min.	25 per cent
Passing 10-mesh sieve and retained on 50-mesh sieve	Max.	25 per cent
Retained on 10-mesh sieve	Max.	1 per cent

For acid-proof mastic the recovered mineral aggregate shall contain not exceeding 5 per cent by weight of material soluble in hydrochloric acid, ascertained by treating 1 g. of the dried filler

with 10 ml. of conc. C.P. HCl, diluting with 50 ml. water, boiling for 15 minutes, filtering through a tared Gooch crucible, washing with hot water, and weighing the residue.

An acid-proof mastic has been proposed, consisting of hard coal-tar pitch and siliceous sand.[128]

Methods of Application. The mastic blocks shall be cracked and broken up into small pieces, weighed, and placed in the mastic kettle or drum of the mastic machine. The proper quantity of flux of suitable grade to give the finished mastic the proper characteristics for the conditions of service to be met, shall then be added. The block and flux shall then be heated together over a slow fire and stirred with iron rods, or by revolving the drum of the machine until the block is entirely disintegrated and the whole mass is fluid and homogeneous. The mineral aggregate, which shall be dry, and if necessary heated beforehand, shall then be added and thoroughly stirred in, and the heating and stirring continued until all particles are thoroughly coated and the mass is of sufficient fluidity to be poured and properly worked into place. The temperature of heating and mixing shall be as low as possible, between 375 and 425° F., and at no time shall exceed 450° F.

If the mastic is to be applied over a wooden floor, one layer of sheathing paper or unimpregnated rag or asbestos felt weighing approximately 5 lb. per 100 sq. ft. shall be laid, lapping the sheets at least 2 in. and nailing as often as is necessary to hold them in place during the application of the mastic. A minimum thickness of 1½ in. mastic shall be used and shall be applied in two layers.

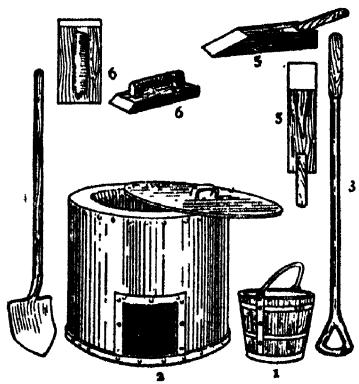
If the mastic is to be applied over concrete, the surface shall be dry, free from sharp projections, dirt, grease, and other foreign substances and shall be properly graded to outlets. The concrete shall not be trowelled, but shall be given a sand-floated finish. The application of an asphalt primer will promote adhesion of the mastic.[129] The hot mixture prepared as above shall be poured on the surface prepared as described, to a uniform thickness of 1 in. It shall then be smoothed out and worked under wooden floats with sufficient pressure until free from voids and blowholes, and a homogeneous layer is obtained. The surface shall then be sprinkled with a mixture of fine, dry, sharp sand containing about 10 per cent of Portland cement and rubbed until a perfectly smooth finish is ob-

tained. The mastic may be surfaced with stone-chips,[130] and it is claimed that a superior finish may be imparted by treating the surface of the mastic, while it is still hot, with an emulsion of ammonium stearate, wax and petroleum distillate, before finishing with the float.[131]

In making joints the hot mastic shall be spread at least 4 in. over the edge of the strip of mastic already in place, so that the cold edge will become soft. The excess material shall then be removed and the edge worked into a perfect bond with the strip being laid. The joints between the strips of the second layer shall not come directly over those in the first layer, which shall be spaced at least 12 in. apart, where the mastic is laid over wooden floors and in other instances where two-layer work is being installed.

A variation consists in applying a mastic base, and while still hot, trowelling on a thinner surface layer composed of a melted mixture of asphalt and a colored pigment.[132]

Where the mastic is to be applied on vertical surfaces, as for instance on the walls of tanks, etc., a wooden or metal form with a slightly curved flare at the bottom shall be used. The side of the form to come in contact with the mastic shall be coated with a solution of soap, talc or other siliceous dust and water to prevent sticking of the mastic.[133] This form shall then be set far enough from the concrete wall or surface to be covered, so as to give a mastic coating of the required thickness. Expanded metal lath shall be used as a reinforcing for the mastic and shall be fastened by any suitable means to the concrete wall, etc. The concrete shall be primed with an asphalt paint, and when the paint has dried to a tacky condition the hot mastic shall be poured into the form and worked and tamped so as to completely flow through the meshes of the lath and form a dense mixture, as free from voids as possible. When the mastic has cooled



1. Steel hooped wooden pail for measuring sand and grit and for carrying hot mixture.
2. Mixing and cooking kettle.
3. Stirrer for agitating hot mass in kettle.
4. Shovel for taking hot mixture from kettle.
5. Spatula or spreading tool.
6. Float or smoothing tool.

FIG. 142.—Tools for Finishing Mastic Floors.

and set, the form shall be removed and the surface of the mastic rubbed smooth with sharp-grained sand.

The tools ordinarily used to lay asphalt mastic are illustrated in Fig. 142. Additional formulas based on the use of mastic prepared from Trinidad asphalt, so proportioned that the completed mixture will carry approximately 12 per cent of asphalt soluble in carbon disulfide, are included in Table LXVI. The use of a small percentage of asbestos fibers is claimed to improve the mixture when used for floorings.[134]

For constructing floors subjected to severe usage, the percentage of asphalt soluble in carbon disulfide may be increased to 17 or 18 per cent by weight, whereupon 60 to 70 per cent by weight of stone chips or other coarse aggregate may be incorporated, having the following granularmetric composition:

Passing $\frac{3}{8}$-in. sieve	50 per cent
Passing $\frac{1}{4}$-in. sieve	25 per cent
Passing $\frac{1}{8}$-in. sieve	25 per cent

Asphalt mastic floors may be reinforced with wire mesh, expanded metal or metal rods.[135] Asphalt mastic is also used in Great Britain and its Colonies for constructing so-called "damp-course" in buildings. It is likewise used as a mortar for cementing together bricks in constructing waterproof masonry,[136] in which case it is trowelled on while hot and plastic.

ASPHALT MASTIC ROOFS

In Europe, asphalt mastic is also used for finishing the roofs of buildings and has given very satisfactory service, even under severe conditions. When used for this purpose, the mastic should be free from silica and magnesia (i.e., it must not contain dolomite). Silician asphalt obtained from the Ragusa deposit is said to give the best results.[137] It is usually fluxed with Trinidad asphalt to the proper percentage of asphalt content. Natural rock asphalts having a limestone base are claimed to be more intimately impregnated with asphalt than is the case with artificial mixtures, although in practice today, ground chalk or limestone is often added. Siliceous matter does not become impregnated with asphalt as does calcium carbonate. Moreover, the mastic may be heated without danger of carbonization in the presence of calcium car-

TABLE LXVI

Varieties of Asphalt Mastic

Proportions by Weight	Foundation	Thickness Recommended, Inches	Mastic		Hard Flux	Soft Flux	Mineral Aggregate					
			Regular	Acid-proof			200-mesh Dust	Sand under 10	Grit ⅛ to ¼	Asbestos	Total Aggregate	
Sidewalks	Concrete	1	33	....	..	6	.....	20	41	....	61	Foot traffic only
Corridors, toilets, stair treads	Concrete	1	40	.....	....	5	.....	15	40	.....	55	One-half hard flux sometimes.
Cellars and storerooms, not cold storage	Concrete	1	45	.....	5	....	.....	15	35	....	50	
Shops (light traffic, etc.)	Concrete	1	50	....	5	.....	....	12	33	.....	45	
Cold storage	Concrete	1	45	....	..	5	.....	15	35	.....	50	
Freight houses, platforms, railroad shops and driveways	Concrete	1½	50	...	5	....	.....	12	33	.....	45	Preferably in two ¾-in. layers.
Cold Storage:												
Bottom course	Wood or Cork	1¼	40	...	....	5	....	20	35	.....	55	Preferably in two layers.
Top course	Wood or Cork	1¼	45	...	....	5	.....	15	35	....	50	
Top course	Wood or Cork	1¼	50	...	.....	4	....	12	34	....	46	If under heavy traffic.
Plating and acid tank rooms:												
Bottom course	Concrete	1¼	...	45	..	5	...	15	35	.....	50	One-half hard flux sometimes.
Top course	or Wood	1¼	.	50	5	...	..	12	33	.....	45	
Reservoirs	Concrete	1½	60	....	..	5	...	5	30	.....	35	Mastic protected with concrete or brick.
Swimming pools:												
Walls and floors	Concrete	1½	60	.....	.	4	.....	5	31	.....	36	Back of and under enameled brick facing.
R. R. bridges (over membrane waterproofing):												
Bottom course	Concrete	1½	60	....	...	5	.....	5	30	.....	35	Laid on paper or dry felt.
Top course	Concrete	1½	50	.....	....	5	.....	12	33	.....	45	Top course mopped with heavy coat of Trinidad asphalt cement and sanded.
As a waterproofing course alone:												
Both courses	Concrete	1½	65	.....	...	5	....	5	25	.....	30	Top protected with concrete or preferably brick with asphalt joints.
Concrete bridges on arches and under pavements	Concrete	1½	55	.....	.....	5	....	10	30	.....	40	Applied on a cold liquid asphalt primer coat.
Magazines, and loading rooms not heavy traffic	Concrete	1	....	....	16	10.3	65.2	....	.....	8.5	73.7	Ungritted mastic.
Fillets and coves	Concrete	1	50	.....	5	.....	.....	45	.....	.....	45	Against walls, columns, etc.
Acid tanks	Concrete	1½	.....	.....	.....	.....	.....	.....	.....	.....		Mixtures, and application vary with conditions.
	Wood	1½	.....	.....	.....	.....	.....	.....	.....	.....		

bonate (limestone), whereas sand or other siliceous matter has a tendency to carbonize the asphalt. The finished mixture should contain approximately 15 per cent soluble in carbon disulfide, showing a penetration of 40 to 50 at 77° F. (Test 9*b*). The following represents a typical analysis of a mixture which has given satisfactory results on roofs: Soluble in carbon disulfide 14.6 per cent; aggregate passing 200-mesh sieve, 35.7 per cent; passing 100-mesh, 9.50 per cent; passing 80-mesh, 2.75 per cent; passing 50-mesh, 10.70 per cent; passing 40-mesh, 6.00 per cent; passing 30-mesh, 8.25 per cent; passing 20-mesh, 7.00 per cent; passing 10-mesh, 4.70 per cent; and retained on 10-mesh 0.80 per cent. The mastic should be applied to roofs in two layers totalling ¾ to 1 in. thick.

Asphalt mastic roofs have given satisfactory service in Great Britain, some of which are over 40 years old, and still free from blemishes.[138] Two such roofs, laid in 1890 ("A") and 1892 ("B") upon analysis (in 1933) were found to be composed of the following constituents:

	Roof "A," Per Cent	Roof "B," Per Cent
Soluble in carbon disulfide	12.7	13.6
Aggregate passing 200-mesh sieve	43.6	41.2
" " 100-mesh "	5.0	5.1
" " 85-mesh "	0.6	0.6
" " 52-mesh "	2.4	2.7
" " 36-mesh "	2.2	1.8
" " 25-mesh "	0.6	0.6
" " 18-mesh "	2.8	2.7
" " 8-mesh "	20.3	19.5
" " ⅛-in. screen	8.8	11.5
" " ¼-in. "	1.0	0.7
Total	100.0	100.0
Asphalt calculated on ungritted material	17.7	18.7
Grit content	28.2	27.5
Penetration of asphalt at 77° F	26	28

It has been proposed to construct asphalt mastic roofs from a mixture of asphalt and coke;[139] also to lay asphalt mastic roofs over sheet metal (e.g., lead);[140] also to surface the roof with a Portland-cement mortar.[141]

BITUMINOUS EXPANSION JOINTS

These are composed of bituminous strips ¼ to 1¼ in. thick, made up with or without a felted or woven fabric or metal rein-

forcement. They are used in connection with concrete or block pavements and installed between the pavement and curbs, also transversely across the pavement to take up the expansive and contractive stresses and strains. When expansion joints are not used, a concrete pavement will crack in cold weather because of the inelasticity of the concrete, and have a tendency to buckle or bulge in hot weather.

Modern practice calls for longitudinal expansion joints along the curbs, and transverse joints spaced at intervals of 40 to 75 ft. The width of the expansion joint should equal the thickness of the pavement (3 to 8 in.).

Four types of bituminous expansion joints are in use, viz.:

(1) *Premolded Strips of High Fusing-point Bituminous Compositions.* These [142] consist of blown asphalt of a high fusing-point (50 parts) mixed with sand (50 parts) and shoddy-dust (25 parts); or of a bituminous mixture extruded through a die under pressure while in a heated state; [143] or kneaded and then rolled on a moving belt; [144] or cast in molds; [145] or pre-cast in the concrete pavement.[146] When cool, the web is cut into joints of the desired size and configuration.[147] An approved mixture consists of grahamite 21 per cent, residual oil derived from non-asphaltic petroleum 49 per cent, and soft native asphalt 30 per cent. The presence of the soft native asphalt enables residual oil derived from non-asphaltic petroleum to flux with the grahamite. The fusing-point of the combination should be in excess of 190° F. (Ring and Ball method). The mixture is claimed to possess the required elasticity, cohesiveness and resistance to temperature changes. In winter, however, the strips must be transported very carefully for they would break into small pieces if allowed to drop.

Longitudinal strips should be ½ in. thick for streets less than 20 ft. wide, ¾ in. for streets 20–30 ft., 1 in. for streets 30–40 ft. and 1¼ in. for streets over 40 ft. wide, placed parallel with, and at each curb line. Transverse joints ¼ to ½ in. thick should be spaced not less than 40 ft. and extend the full width of the brick, wood, stone-block or concrete pavement.

A proprietary product composed of gilsonite fluxed with blown petroleum asphalt tests as follows:

(Test 9*b*)	Penetration at 115° F.	50–60
	Penetration at 77° F.	35–40
	Penetration at 32° F.	20–30
(Test 15*b*)	Fusing-point (R. and B. method)	140–255° F.
(Test 21)	Soluble in carbon disulfide	99.5 per cent

The Commission of Highways of the State of New York specifies that the premolded joint shall be as follows:

(Test 7)	Specific gravity at 77° F.	0.98–1.05
(Test 9*b*)	Penetration at 115° F.	Less than 45
	Penetration at 77° F.	15–35
	Penetration at 32° F.	Greater than 12
(Test 15*b*)	Fusing-point (R. and B. method)	220–250° F.
(Test 16)	Volatile matter at 325° F., 5 hours	Less than 1 per cent
(Test 21)	Soluble in carbon disulfide	Greater than 98.5 per cent
(Test 22)	Soluble in carbon tetrachloride	Greater than 99.8 per cent
(Test 23)	Soluble in 76° petroleum naphtha	50–75 per cent

A modification consists in commingling felted fibers with the bituminous matter in the presence of water, by forcing them through a perforated plate, and after evaporating the water, molding the mixture to form the joint.[148]

The following fillers have been suggested for incorporation in the asphalt:

Granulated Matter.

(*a*) *Organic:* Including granulated cork;[149] granulated corn cobs;[150] cottonseed hulls.[151]

Fibrous Matter.

(*a*) *Organic:* Including sawdust;[152] excelsior;[153] cocoanut fibers;[154] straw;[155] cotton stalks;[156] flax, hemp or sisal fibers;[157] yucca (sapweed) fibers;[158] Tampica fiber;[159] latex, rubber, shredded rubber-bearing shrubs, or shredded rubberized fabric;[160] shredded rags;[161] shredded roofing scrap;[162] animal hair or wool;[163] also any of the fibers referred to in Chapter XXIV (Group C-β).

(*b*) *Inorganic:* Including asbestos or mineral wool.[164]

(*c*) *Mixtures of Organic and Inorganic:* Including asbestos and vegetable fibers;[165] infusorial earth and vegetable fibers;[166] mineral filler and vegetable fibers;[167] papier maché and flake mica;[168] etc.

Sundry Mixtures.

Including: asphalt, fibrous matter and a dispersing agent;[169] asphalt, fibrous matter, starch paste and glycerin;[170] asphalt, fibrous matter and a vegetable oil;[171] sponge asphalt (i.e., cellular asphalt), with or without fibrous matter;[172] etc.

To prevent adhesion in transit, or to improve the wearing properties of the expansion-joint, it may be surfaced with any of the following, viz.: hard asphalt;[173] wax;[174] rubber or rubber latex;[175] a slow-drying adhesive substance;[176] fibrous matter (e.g., asbestos);[177] Portland cement;[178] sodium silicate;[179] etc.

(2) *Joints Composed of a Thick Layer of Bituminous Material*

Reinforced on One or Both Surfaces with Webs of Plain or Bituminized Fabric. This type of expansion joint is composed of a relatively thick layer of bituminous composition similar to the foregoing, reinforced with one layer of paper,[180] or two layers of plain or bituminized felted fabric,[181] or woven fabric impregnated with asphalt,[182] or two layers of open-mesh burlap,[183] or two layers of perforated asbestos felt,[184] a core of felted fibers,[185] etc.

The function of the fabric is to strengthen the joint, and enable it to be transported in cold weather without danger of fracturing, thus overcoming the objection against the foregoing type. The bituminous layer in the center should have a high fusing-point (above 185° F., K. and S. method) and preferably a low susceptibility index. It may be composed of asphaltic constituents, sometimes mixed with 25 to 50 per cent of finely divided mineral matter (calcium carbonate, shale, clay, silica, fuller's earth or slate), or from 15 to 30 per cent by weight of ground wood ("wood flour") or fibrous matter. The function of the filler is to increase the toughness and resistance of the mixture to temperature changes and in certain cases to reduce the cost.

The joint is manufactured either by pouring the melted bituminous material into a receptacle having a sheet of plain or impregnated fabric at the bottom, or it may be manufactured continuously on a form of machine used for turning out prepared roofing, in which the layers of fabric are fastened together by introducing the melted or plastic bituminous composition in between, carefully adjusting the thickness of the assembled sheet to correspond with that of the joint desired. The width of the sheet should similarly correspond to the length of the completed joint (usually 6 ft.). When the assembled sheet has cooled, it is cut transversely with mechanically actuated knives, into strips 3 to 6 in. wide, depending upon the thickness of the pavement for which they are intended.

(3) *One or More Layers of Bituminized Fabric the Latter Cemented with a Bituminous Adhesive.* One form of paving strip is composed of a single layer of tarred felt,[186] or fibrous board saturated with asphalt;[187] or woven fabric saturated and coated with asphalt, having particles of cork or sawdust embedded in the surfaces;[188] or saturated and coated felt embossed in the form of projections and indentations;[189] or two or more layers of asphalt-saturated felt cemented together with an asphaltic adhesive;[190] or saturated fabric enclosed between two layers of bituminized felt, cemented together with asphalt;[191] or a layer of fibrous board (which may or may not be saturated) surfaced on both sides with asphalt-saturated felt.[192] The strip when assembled varies from ¼ to ¾ in. thick, and the raw felt sheets range from No. 50 to No. 75 on the felt marker's scale.

In manufacturing this form paving joint, one or more webs of felted fabric are first impregnated individually with melted asphalt, usually quite soft in consistency (fusing-point 90–140° F., K. and S. method), and are then joined by comparatively thin layers of a harder asphaltic mixture (fusing-point 140–180° F., K. and S. method). The outer surfaces are not coated. The width of the web corresponds to the length of the paving joints (usually 6 ft.). After the webs are sealed together and allowed to cool, they are cut into strips corresponding to the thickness of the pavement. Another variation involved the use of wood-pulp corrugated under pressure and impregnated with asphalt or tar,[193] or a core of wood-veneer saturated with asphalt and coated on both sides.[194]

(4) *Armored Bituminized Fabric.* Another type of paving joint adapted solely for concrete roads, known as an "armored joint," consists of a layer of bituminized fabric reinforced on either side by a thin metal plate.[195] The joint may either be flat or corrugated,[196] and the upper part constructed with flanges or projections to protect the edges of the adjacent concrete sections.

Another modification is composed of a strip of high fusing-point bituminous composition (similar to Type 1) reinforced in the center with a metallic core,[197] or reinforced with strips of wood.[198] Still another consists of a strip of high fusing-point bituminous composition protected on the surface with a trough-shaped shell of wire mesh, to which in turn paper or cloth is cemented fast.[199] Another involves the use of a plurality of transverse dowels.[200]

Bituminous expansion-joints may be fastened at the ends with metal staples;[201] or anchored to the concrete pavement by means of transverse wooden or metal dowels;[202] or embedded below the surface of the concrete;[203] or the sides of the joint may be depressed in zones to cause it to key with the concrete.[204]

BITUMINOUS RAIL-FILLERS

These consist of a premolded bituminous composition *A*, surfaced on one side with a layer of saturated felt *B*, as illustrated in Fig. 143.[205] They are intended for use alongside the rails of traction lines where these abut any of the various types of bituminous pavements, forming a cushion between the two, and thereby preventing the disintegration of the pavement alongside the rail, under the impact of traffic. The bituminous composition is similar to that used in Type 1, and usually contains fibers intermixed throughout.

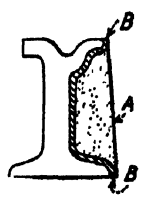


FIG. 143. — Rail-filler.

ASPHALT BRIDGE-PLANKING

This consists of premolded bituminous compositions, similar to Type 1 described under "Bituminous Expansion Joints," shaped in the form of planks 2 to 3 in. thick, and intended for paving bridges or culverts. The bridge-planking is cemented to the bridge roadway by means of a bituminous grout applied in a melted condition. Interlocking forms of bridge-planking have also been recommended.[206]

BITUMINOUS REVETMENT

Revetment strips for protecting the banks or levees or sea-walls of streams and rivers to prevent soil erosion, are composed of a bituminous mixture, similar to bituminous concrete, in some cases reinforced with metal or other fabric.[207] These are made in continuous lengths on a specially equipped barge adjacent to the bank, from which the revetment is run out and anchored to the earth in a suitable manner.

CHAPTER XXIX

BITUMINIZED FABRICS, FELTS AND PAPERS FOR ROOFING. FLOORING, WATER-PROOFING, BUILDING AND INSULATING PURPOSES

PREPARED SHEET ROOFINGS

These are composed of a single layer, or a plurality of layers assembled together, each composed of a woven or felted fabric, saturated, coated, or both saturated and coated with bituminous compositions, and in special cases reinforced with metal. The finished structure may be supplied in flat sheets or wound up in rolls of suitable length and width. These fabrics and bituminous compositions may be assembled in innumerable combinations.[1]

Felted Fabrics. These are generally formed of rag or asbestos fibers, with or without additions, on a machine similar to that used for manufacturing paper. If rags are employed, they are first run through a series of revolving knives known as "cutters," which shred them into small fragments, and then run into the "beaters," where they are ground into a pulp with water. A beater separates the strands forming the cloth into the individual fibers without materially shortening them. A charge is beaten one-half to three hours until all the lumps have been broken up, whereupon the "pulp" is passed through a "screen" to remove any foreign particles, and then run on the felt machine (Fig. 144), where it is formed into a sheet of predetermined thickness. Cylinders 1 and 2, covered with fine wire cloth, are partly immersed in the tanks *T–T* containing the beaten fibers suspended in water. As the cylinders revolve, the wire cloth acts as a strainer and picks up a layer of fibers, letting the water run to waste. The machine is equipped with three endless webs of cloth, 3, 5 and 16. Cloth 5 is first pressed in contact with cylinder 1 by the couch roll 11, causing the fibers to adhere to the cloth. Cylinder 2 similarly adds a layer of fibers under pressure of the couch roll 12. The suction box

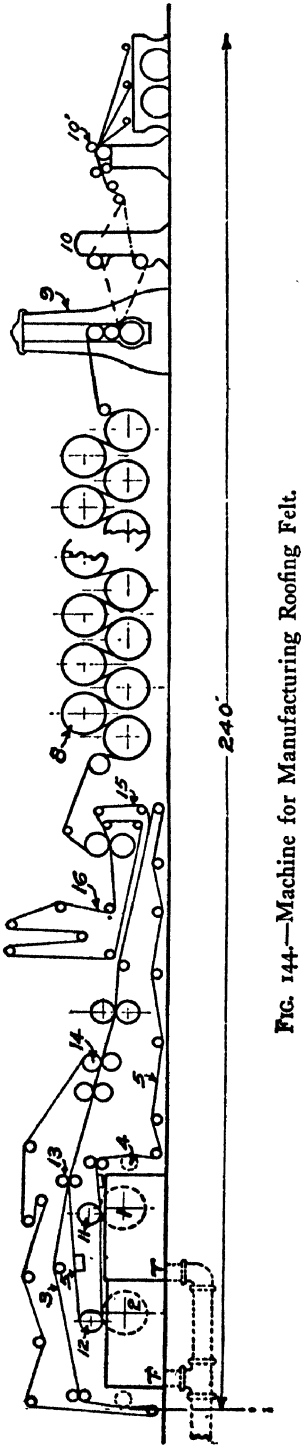


FIG. 144.—Machine for Manufacturing Roofing Felt.

shown alongside rollers 11 and 12 draws the surplus water from the newly formed sheet of felt, which then passes between cloths 3 and 5, where it is subjected to gradually increased pressure by the rolls 13 and 14. At roll 14, the upper cloth 3 returns, while cloth 5 carries the sheet of felt to 15, where the felt now just strong enough to hold together, leaves cloth 5 and passes between pressure rolls against the third cloth 16. The felt is now carried over a series of steam rolls 8, of which modern felt machines are equipped with 75 to 150. These expel the remaining moisture, whereupon the sheet is given a smooth surface by the calendar rolls 9, and wound into rolls at 10, while at 10′ the wide sheet is slit into narrower sheets either 32 or 36 in. wide, and automatically rewound into rolls by the winders shown at the extreme right.

Rag-felt

The Felt Manufacturers' Association [2] of the United States recognizes six classes of raw materials for making roofing felt, viz.:

No. 1, *Roofing Rags.* These constitute soft rags carrying a percentage of wool, and include satinet garments, men's coats, pants, vests, mixed linsies, women's coats, sacks and cloth skirts, all containing a portion of wool fiber; excluding overalls, khaki, canvas, duck, quilts, seams and strippings or similar materials.

No. 1-*S*, *Seams and Cloth Strippings from Garments.*

No. 2, *Roofing Rags.* These consist of cotton rags, and include large and small cotton rags, linings, rag carpets, print rags and stockings, excluding silk rags, rayon and wood-fibered rags.

No. 3, *Jute Bagging.* These include

bags and sacks free from fertilizer, charcoal, coal, cement, chemicals of all kinds, lime, plaster, salt and all foreign substances, and exclude jute waste and jute droppings and all foreign substances of whatsoever nature or description.

No. 4, Carpets. These include Brussels and hardback carpets, also upholstering and plush cuttings.

No. 5, Roofing Rags. This class includes: (*a*) tailor rags free from all rubbish and paper; (*b*) tailor rags containing not over 10 per cent paper; (*c*) tailor rags containing over 10 per cent, but not exceeding 50 per cent of paper.

No. 6, City Dump Rags. Including rags of all sorts, and of variable composition.

Miscellaneous Materials. These are also used for manufacturing felt, and include window shades, cement sacks, jute waste, jute droppings, carpet waste, soft strings, cotton- and wool-filled mattresses or quilts, buckram, cotton linters, cotton-seed hull fiber and wool waste. The permissible amount of moisture in the rags shall not exceed 9 per cent by weight.

The following materials are distinctly excluded by the Association, viz.: paper shall be absent except in No. 5 "Roofing Rags," classes (*b*) and (*c*); likewise the following are unacceptable in all classes: rubbish, shoe cuttings, felt boots, hats, corsets, suspenders, oil cloth, matting, leather, rubber, rope, mackintosh clippings, pasted stock, wool dust, wood, stones, metal of all kinds, tin cans, glass, ashes, bones, excelsior, oily rags, canvas, sisal, pickers, fertilizer and charcoal bags, coal and cement sacks, chemical-, salt-, lime- and plaster-bags, silk rags and silk stockings, rayon, buffing wheels, rubberized cloth, auto curtains and cushions, dirt, felt punchings and trimmings, etc.

The following fibers are present in the foregoing classes of materials:

True rag fibers, including: (1) cotton and linen fibers; (2) wool and silk fibers; (3) jute and manila fibers.

Paper fibers, including: (1) mechanical (i.e., ground) wood pulp; (2) chemical wood pulp (sulphite and sulphate).

No. 1 rags, contain cotton (and linen) also a percentage of wool (and silk) fibers.

No. 2 rags, contain mostly cotton fibers.

No. 3 rags, contain cotton, also a percentage of jute and manila fibers.

No. 4 rags, contain mostly jute and manila fibers.
No. 5 rags, contain cotton and wool with or without paper fibers.
No. 6 rags, contain a mixture of all the fibers present in the previous classes.

The various classes of rags are mixed in suitable proportions in the manufacture of roofing felt. Classes 1 and 2 tend to "soften" the felt, and open up its pores thereby enabling it to absorb a larger percentage of bituminous saturation. Classes 3, 4 and 5 tend to "harden" the felt and make it less pliable, less absorbent and denser. Class 6 acts variably, depending upon the composition of the rags used. It is customary to mix Classes 1 and 2 with Classes 3, 4, 5 and 6 in proportions ranging from 5 : 1 to 1 : 2, depending upon the nature of the rags available, and the quality of the felt to be manufactured.

Classification of Foreign Rags. These are grouped into the following classifications:

Linsey garments, including soft rags such as satinet garments; men's coats, pants and vests, women's coats and cloth skirts.
Dark cottons, including large and small cotton rags, e.g., linings, print rags and cotton stockings.
No. 1 shopperies, including clean tailor cuttings only.
No. 2 shopperies, including clean tailor rags, which may contain paper to an amount specified.

A *high-grade* roofing felt made up approximately of 75–80 per cent "soft" rags and 20–25 per cent "hard" rags, will show the following composition on examining the finished sheet microscopically (Test 85):

Cotton fibers	50–70 per cent
Wool fibers	10–20 per cent
Jute and manila fibers	5–15 per cent
Wood fibers (paper)	1–5 per cent

Roofing felt is often spoken of as "wool felt," but this term is somewhat of a misnomer, since no roofing felt is composed *entirely* of wool fibers. It is impractical to manufacture roofing felt from wool fibers alone, as they are so soft and fluffy that they will not form a satisfactory sheet on a felt machine. It is necessary to have other fibers present, to produce a strong, compact sheet, not to break on the calendar rolls or dryers, and to absorb the proper percentage of bituminous saturation afterwards.

The durability of the various fibers is in exact proportion to their prices. Their comparative costs, taking wool as 100, are approximately as follows:

Wool	100
Cotton	60
Jute and manila	40
Paper (including mechanical and chemical wood fibers)	20

Wool fibers are most durable and least affected upon contact with moisture or the sun's rays. Nature provides wool for covering animals to protect them against exposure to the elements. Cotton fibers come next in durability. It is significant that cotton has always been used for making sail-cloths and covering porches or decks of steamers, exposed to the weather. Most of us have observed how much more rapidly jute and manila fibers, as for example in the form of burlap bagging, will decay, than cotton or wool. Paper fibers require little comment. Everyone knows that a newspaper soon falls to pieces when left out-of-doors in the sun and rain, chemical wood fibers (sulphite and sulphate) being somewhat more weather-resistant than mechanically ground wood-pulp.[3]

The particular kind of fiber present does not influence the strength of the felt, which is controlled largely by the following factors:

(1) The *length* of the fibers in the felt. This is predetermined by:

(*a*) The length of the fibers, as they existed in the rags.
(*b*) The extent to which they have been broken up and shortened in the beaters.

(2) The extent to which the rag or paper stock has undergone eremacausis. Old rags or papers which have been allowed to rot before being converted into felt will produce a weaker sheet than when new rags or paper are employed.

(3) The skill displayed in "running" the sheet on the felt machine.

Many substitute fibers have been suggested for manufacturing roofing felt, including the following:

1. *Organic Fibers*:

Waste papers, ground wood;[4] soda pulp;[5] sulfite and kraft screenings;[6] α–cellulose or α–pulp (produced by cooking wood pulp with a mixture of NaOH and Na_2S);[7] chemical wood pulp

and sawdust;[8] wood fibers cooked with an alkaline polysulphide;[9] hydrolyzed wood waste (i.e., sawdust cooked with sulfuric acid);[10] wood lignin;[11] shredded bark of trees (e.g., redwood-, oak- or tan-bark);[12] straw fibers;[13] sugar-cane fibers (i.e., bagasse);[14] cocoanut fibers;[15] disintegrated bamboo;[16] sea-grass and sea-weed;[17] moss and peat;[18] rags and kapoc fibers;[19] rag fibers treated with alkali;[20] bast flax fibers cooked with alkali;[21] corn-stalks cooked with alkali;[22] cordage cooked with alkali;[23] reeds cooked with alkali;[24] beet-pulp cooked with alkali;[25] disintegrated leather;[26] leather fibers and animal hair;[27] cotton linters and animal hair;[28] cotton rags, animal hair and kraft pulp;[29] ground up sponges;[30] bituminized sawdust fibers;[31] macerated worn-out automobile tires;[32] etc.

2. *Inorganic Fibers, etc.*:

Slag-wool or mineral wool;[33] asbestos; glass wool;[34] infusorial earth;[35] talc;[36] clay; powdered charcoal;[37] etc.

3. *Combinations of Inorganic with Organic Fibers*:

Asbestos with rag fibers;[38] asbestos with wood-pulp;[39] etc.

Waste paper is used most extensively as a substitute for rag stock. It does not appreciably affect the strength of the felt, but it tends to increase its density, reduce the percentage of bituminous saturation absorbed, make it harder and less pliable, and also less weather-resistant. The author has examined samples of roofing felt containing as much as 85 per cent of paper in admixture with rag stock.

If properly made, felt should not carry more than 8 per cent by weight of mineral ash. The moisture content as furnished by the manufacturer should not exceed 5 per cent by weight, whereas upon storage in a humid atmosphere the felt will absorb more moisture until an equilibrium is reached. Thus in air at room temperature saturated with moisture, the felt will absorb a maximum of 20 per cent, whereas in air carrying 65 per cent humidity, the felt will absorb up to 8.5 per cent.

Rag felt is marketed on the basis of its weight in pounds per 480 sq. ft., known as the "number," ranging from 17 to as high as 90. The "number" of the felt multiplied by 0.225 will give its weight in pounds per 108 sq. ft., and when multiplied by 0.208 will give its weight per 100 sq. ft. High-grade rag felt will test approximately 1 mil in thickness (Test 78), and not less than 0.5 lb. on the Mullen strength tester (Test 79), per unit "number." These relations hold approximately constant for all weights.

The following standards have been established:[40]

(1) Rags, fibrous textile waste, and waste paper may be used,

but the direct addition of wood pulp, straw pulp, peat, sawdust and mineral loading material is forbidden.

(2) The felt must not yield more than 12 per cent of ash.

(3) The moisture content of the air dry felt should not exceed 12 per cent.

(4) Felt when immersed in anthracene oil should not absorb less than 120 per cent by weight.

(5) Felt of normal thickness (at least 400 grams per square meter) should have a breaking weight [41] of at least 3 kilos (for a strip 15 mm. wide) in the longitudinal direction.

The use of a perforated sheet of felt has been suggested to effect a saving in weight,[42] and which when saturated and coated with bituminous substances will have the perforations completely closed. It is claimed that similar results may be obtained by cutting holes in the sheet and leaving the burrs or projections protrude from the surface; [43] or else by "napping" the surface of the dry felt, so that when it is saturated an excess of the saturant will be retained on the surface and gradually work its way into the center of the sheet; [44] or else by indenting the surface of the saturated sheet before it is coated.[45] A method has been proposed to make a sheet of variable thickness, so that when severed into units, it will be tapered and thus adapted to making tapered shingles.[46] Methods have been proposed to increase the strength of the felt, or stiffen it, by sizing with viscose solution (cellulose xanthate),[47] or with an emulsion of starch paste and bentonite.[48] To facilitate testing the felt for the amount of saturant it will absorb, a method has been proposed which consists in manufacturing the sheet with lateral extensions intended to be removed and used for testing purposes.[49]

Methods of Fireproofing Rag-felt. Processes have also been suggested for fireproofing the felt, by treating it with various inorganic fireproofing agents prior to its impregnation with the bituminous saturant, among which may be mentioned: [50] sodium tungstate; [51] borax or boracic acid; [52] calcium borate; [53] alum; [54] soluble silicates (e.g., sodium silicate); [55] ammonium-magnesium phosphate; [56] sodium phosphate, ammonium phosphate or zinc-ammonium phosphate; [57] phosphoric acid, with or without aluminium acetate; [58] ammonium chloride; [59] calcium chloride; [60] zinc chloride and ammonia; [61] magnesium chloride or ammonium chloride with borax and alum; [62] stannic chloride or sodium stannate followed by am-

monium sulfate;[63] zinc chloride and sodium silicate;[64] titanium or zirconium sulfate;[65] sodium molybdate;[66] ferrous oxalate;[67] tan-bark extract followed with alum;[68] magnesium sulfate, ammonium sulfate and dextrine, or magnesium chloride, ammonium chloride and dextrine;[69] treating bituminized sheets with an aqueous solution of an inorganic fireproofing agent in the presence of a wetting-out agent (e.g., a sodium salt of naphthalene sulfonic acid);[70] soda and salt;[71] magnesium chloride, borax and potassium dichromate; borax and potassium bitartrate; ammonium toluene sulfonate and ammonium borate; zinc chloride, borax and ammonium bromide; ammonium toluene sulfonate and ammonium chloride;[72] etc.

String-felt or Threaded-felt. This is made in the same manner as rag-felt, but with threads or strings introduced in continuous lengths, in a longitudinal direction of the sheet, while it is formed on the felt machine.[73]

Asbestos-felt.[74] This is made on substantially the same type of machine as rag-felt. It is sometimes made from asbestos fibers alone,[75] but when intended for saturating purposes it is generally composed of a mixture of asbestos with not exceeding 15 per cent by weight of animal hair[76] to open up its texture, or a proportion of cotton fibers (e.g., cotton linters) may be added to accomplish this purpose. In all cases it is customary to add a binder consisting of starch paste, glue, dextrine, or aluminum silicate.[77]

The varieties of asbestos fibers include the following principal groups:[78]

	Serpentine Group	Amphibole (Hornblende) Group
Mineralogical Names	Chrysotile (Canada, Rhodesia, South Africa, Russia and Australia) $Mg_3(SiO_3)_2 \cdot 2H_2O$	Crocidolite (blue asbestos; South Africa) and Amosite (yellow asbestos; South Africa) $NaFe_2(SiO_3)_3 \cdot H_2O$ Tremolite (Italy and Russia); Anthrophyllite (Maryland) and Actinolite (U. S. A.) $(MgFe)_3Ca(SiO_3)_4$
Water of hydration	13 to 15 per cent	2 to 5 per cent
Color	White to yellowish	White to grey to brown, also blue.
Character of fibers	Soft and fine.	Harsh and springy.
Temp. of dehydration	400 to 850° C.	300 to 600° C.
Resistance to acids	Poor.	Good.

Asbestos-felts are marketed on the basis of weight in pounds per 100 sq. ft., ranging from 6 to 50 lb. Laminated sheets have also been suggested, composed of a layer of asbestos fibers in juxtaposition to a layer of rag fibers.[79]

Mixtures of asbestos fibers, mineral wool and colloidal clay have also been proposed.[80]

Woven Fabrics. The woven fabrics ordinarily used for manufacturing prepared roofings include burlap or hessian (composed of jute fibers), drill [81] and duck (composed of cotton fibers). These are marketed in various weights, expressed in arbitrary ways.[82] Woven fabrics do not take up nearly so large a percentage of bituminous saturation as felted fabrics. Burlaps [83] are designated by weight in ounces per lineal yard 40 in. wide, and 7½, 8 and 10 are generally used for roofing and waterproofing purposes. "Regular" or "ounce" ducks are designated by the weight in ounces per lineal yard 29 in. wide, and 6 to 15 are generally used for the foregoing purposes. There are three grades or qualities of "ounce" ducks, viz.: "U. S. Army" standard duck, made of multiple-ply yarns both in warp and filling; "double-filled" duck made of single-ply yarns in the warp and multiple-ply in the filling; and "single-filled" duck made of single-ply yarns in both warp and filling. Another type of duck is known as "enamelling" duck, which is designated in numbers arbitrarily from 0 (or more ciphers) to 12. Number 00, the heaviest commonly used, weighs 20 oz. per lineal yard 22 in. wide, 0 weighs 19 oz., 1 weighs 18 oz., and each number greater than 1 represents a decrease of 1 oz. per lineal yard 22 in. wide. They are generally manufactured in widths ranging from 22 to 144 in. and are composed of multiple-ply yarns in both warp and filling. Numbers 8 to 12 are usually used for manufacturing roofing products. The use of a woven fabric in which the warp consists of metallic fibers and the filling of jute or wool has also been proposed.[84]

Bituminous Saturating Compositions. Bituminous materials suitable for impregnating felted or woven fabrics are usually soft in consistency at room temperature, ranging from semi-liquids to semi-solids.

The following classes of bituminous substances have been used for this purpose:

Asphaltic Products,[85] including soft native asphalts, residual oils, soft residual asphalts, soft blown petroleum asphalts, blown asphalts having a fairly high fusing-point;[86] pressure tars and soft sludge asphalts; fatty-acid pitch; sulfurized fatty-acid pitch;[87] wax tailings;[88] wool grease;[89] etc., used either singly or in various combinations when of the proper consistency, or if too soft and liquid they may be hardened by combining with suitable proportions of pure native asphalts, residual asphalts, blown petroleum asphalt; sludge asphalt, or animal and vegetable oils and fats. For example, mixtures have been proposed composed of asphalt fluxed with linseed oil;[90] cottonseed oil;[91] castor oil;[92] linseed or china-wood oil with dryers;[93] vegetable oil (e.g., linseed, cottonseed, castor, rapeseed, palm oil, etc.) heated in the presence of nitric acid, sulfur or sulfur dichloride;[94] animal or vegetable oil heated in the presence of oxygen and a catalyst (e.g., Ni or Mn resinate or naphthenate);[95] fish oil;[96] oleic or stearic acid and β-tetrahydronaphthalene;[97] etc.

Tar Products,[98] including coal tar,[99] water-gas tar and oil-gas tar, used either singly or in various combinations, or tempered to the proper consistency with the corresponding pitch obtained by distilling the tar to expel the highly volatile oils.

The tar products are used for manufacturing single-layered tarred felt for constructing "built-up" roofings and waterproofing membranes, also for manufacturing multiple-layered prepared roofings. They are not recommended for producing single-layered prepared roofings, due to the fact that they are insufficiently weather-resisting. On the other hand, the asphaltic products, because of their greater weather-resistance may in general be used indiscriminately for manufacturing single or multiple-layered roofing and waterproofing products.

At the present time, the saturants most commonly used include soft coal-tar pitch, residual oil, soft residual asphalt and soft blown petroleum asphalt. Mixtures of tars and asphaltic products have also been suggested;[100] likewise a saturant having a penetration at 77° F. (Test 9*b*) of not exceeding 11;[101] hard coal-tar pitch fluxed with rosin oil;[102] mixtures of asphalt or coal-tar pitch and rosin;[103] asphalt fluxed with paraffin oil;[104] asphalt and celluloid;[105] mixtures containing rubber;[106] paraffin wax;[107] etc.

Saturating mixtures prepared from asphaltic products should comply with the following characteristics:

(1) The viscosity at the saturating temperature should be as low as possible to accelerate the speed of absorption by the fabric.

(2) The penetration at 77° F. should be greater than 40 (Test 9*b*), and consistency (Test 9*c*) less than 15, otherwise the saturated fabric will be brittle.

(3) The susceptibility index should be as low as possible, and preferably less than 30.

(4) The saturant should be ductile.

(5) Its fusing-point by the R. and B. method (Test 15*b*) should range from 100 to 160° F. The saturants for felts and roll-roofings run between 100 and 140° F., and those for shingles between 140 and 160° F.

(6) It should contain the smallest possible percentage of volatile matter, and preferably less than 3 per cent at 500° F. in five hours (Test 16*a*).

(7) It should have a flash-point well above the temperature to which it is raised during the process of fluxing or while saturating the fabric (at least 50° F.), and preferably above 410° F. (Test 17*a*).

(8) It should contain the largest possible percentage soluble in carbon disulfide and preferably exceeding 98 per cent.

(9) It should contain a comparatively high percentage soluble in 88° petroleum naphtha, and preferably exceeding 85 per cent.

(10) It should be weatherproof.

Light-colored saturants have also been proposed, consisting of rosin or rosin oil;[108] pine tar;[109] rosin and linseed oil;[110] rosin and paraffin oil;[111] fatty-acid pitch, rosin and linseed or paraffin oil;[112] asphalt combined with paraffin wax and vaseline or stearine;[113] castor or other non-drying vegetable oil;[114] also mixtures of coal tar with collodion.[115] Similarly, colored saturants have been recommended, composed of a fat-soluble aniline dye dissolved in a mixture of asphalt and vaseline,[116] or in a mixture of asphalt, rosin and linseed oil;[117] likewise a mixture of wax tailings with a fat-soluble dye or a colored mineral pigment;[118] the expedient of first dyeing the felt and then saturating same with a light-colored saturant (e.g., wax tailings);[119] the use of a mineral oxide pigment admixed with the bituminous saturant;[120] saturating with a mixture of cumarone resin, nitrocellulose, a vegetable drying oil and a mineral pigment;[121] applying a mixture of fibers, water, mineral pig-

ments and an aqueous bituminous-clay dispersion to the surface of the felt as it is formed on the felt machine;[122] etc.

Various fire-resisting compositions[123] have been proposed from time to time for use in connection with saturants as well as coatings, including: chlorinated bituminous substances (e.g., coal tar, naphthalene, dead oil, chlorinated di- or poly-phenyl dissolved in the asphalt,[124] wax tailings, etc.);[125] brominated bituminous substances (e.g., dead oil);[126] dissolving antimony or bismuth trichloride or tri-iodide in the saturants;[127] dissolving triphenyl or tricresyl phosphate in the saturant;[128] first dissolving casein in sulfonated castor oil, which in turn is mixed with the saturant and then coagulated with formaldehyde, thereby producing a colloidal dispersion of casein;[129] mixing the bituminous saturant with inorganic fireproofing agents, such as borax and ammonium sulfate;[130] sodium phosphate or tungstate;[131] calcium chloride;[132] potassium bi-tartrate or metallic tartrates;[133] ammonium phosphate or boric acid in admixture with chlorinated naphthalene;[134] etc. The flame-retarding properties of fatty-acid pitch has also been observed.[135]

Bituminous Coating and Adhesive Compositions. These may be used for surfacing the saturated fabrics (single or multiple layered) and also for cementing together two or more layers of fabric (all of which may be bituminized, or some bituminized and some untreated) in manufacturing multiple layered sheets. The coating and adhesive compositions include the same groups of substances used for the saturant, but prepared of a harder consistency and usually also of a higher fusing-point. The bituminous materials first used for these purposes included the native asphalts,[136] sludge asphalts,[137] residual asphalts,[138] fatty-acid pitches,[139] fluxed gilsonite,[140] wurtzilite asphalt,[141] etc. The addition of vegetable or animal oils impart weatherproof properties; likewise the admixture of vegetable oil and sulfur.[142]

A method has been suggested to apply an emulsion of asphalt, colloidal clay and water, with or without the addition of fibers, whereupon the water evaporates, leaving an insoluble coating of asphalt;[143] or this same composition may be surfaced with mineral granules;[144] or an aqueous clay dispersion of fatty-acid pitch containing a mineral pigment, with or without a surfacing of mineral granules;[145] etc. Another procedure consists in applying two super-

imposed coatings, one of coal-tar pitch, followed by a surface-coating of asphalt.[146]

The addition of rubber has been proposed, but rubber is decidedly lacking in weatherproof properties and consequently detracts from the weather-resistance of the mixture. The following compositions, however, have been recommended, viz.: asphalt and rubber;[147] coal-tar pitch and rubber;[148] asphalt, rubber and mineral fillers;[149] coal-tar pitch, rubber and mineral fillers;[150] asphalt, fatty-acid pitch and rubber;[151] mixtures containing rubber latex;[152] phenol-aldehyde resin and vegetable drying oils;[153] etc.

The main requisite of the bituminous coating composition is that it should possess maximum weather-resisting properties, since the longevity of the prepared roofing depends largely upon the unalterability and integrity of the coating. The principles to be followed in making this mixture have already been discussed.

Bituminous coating and cementing compounds should preferably comply with the following characteristics:

(1) The appearance of the surface on aging (Test 3) should be bright, and especially where the bituminous coating is to be surfaced with talc or other white mineral dusting finish.

(2) The hardness at 77° F. should fall between 10 and 50 on the penetrometer (Test 9*b*), and between 15 and 30 on the consistometer (Test 9*c*).

(3) The susceptibility index should be as low as possible, and in the case of asphaltic compounds preferably less than 35.

(4) The ductility should preferably be greater than 1 at 77° F. (Test 10*b*).

(5) The tensile strength should preferably be greater than 0.5 at 77° F. (Test 11).

(6) The fusing-point should be such that the composition will not be affected by the highest sun temperature to which it may be subjected. It should not be lower than 175° F., or higher than 260° F. by the Ring and Ball method (Test 15*b*), the upper limit applying to products having a low susceptibility index, including blown petroleum asphalts, fluxed asphaltites, etc.; and the lower limit to coatings surfaced with fine sand or coarsely ground talc. The corresponding range by the K. and S. method (Test 15*a*) shall be between 165 and 240° F.

(7) The volatile matter should preferably be less than 2 per cent at 500° F. in 5 hours (Test 16).

(8) The flash-point should preferably be above 410° F. (Test 17*a*).

(9) The solubility in carbon disulfide should preferably not be less than 98 per cent, although this is subject to modification where mineral ingredients are purposely added for the reasons which follow.

(10) The larger the percentage of fatty substances (saponifiable) present in the mixture, the better will be its weather-resisting properties, other things being equal.

The presence of solid paraffins (Test 33) is considered objectionable [154] for the following reasons:

(*a*) In time, it will sweat to the surface, forming a dull, greasy film.

(*b*) It makes the substance more susceptible to temperature changes, i.e., harder and more brittle at low temperatures, also softer and more fluid at high temperatures; and likewise lowers its viscosity when melted.

(*c*) It serves to decrease the adhesiveness and cohesiveness of the product.

(*d*) It affects the stability of the mixture on continued heating or repeated heatings, under which circumstances the paraffin assumes a crystalline form. It has been observed that the addition of sulfur will inhibit the crystallization of paraffin, by acting as a protective colloid, and will prevent the lowering of the ductility of the product on protracted heating.

As far as the writer is able to determine, the presence of moderate amounts of solid paraffin (i.e., up to 4.5 per cent) exerts no marked influence upon the weathering properties of the mixture.

Fillers. It is sometimes customary to mix powdered or fibrous substances of either inorganic or organic origin with the melted bituminous constituents of the coating, before they are applied to the surface of the roofing, for the purpose of reducing the cost of the finished product, or to increase its weather-resistance, or to impart a color to the surface-coating. Many substances have from time to time been recommended for this purpose. At the present time the most popular fillers include powdered silica (sand), limestone, slate dust, clay, etc. These serve primarily as extenders and have the object of reducing the cost of the mixture, although in some cases, particularly when mineral fillers of an extreme degree of fineness are incorporated, the weather-resistance of the mixture is improved

materially. Another procedure consists in applying part of the bituminous coating first, then the mineral filler, and finally the balance of the bituminous matter, thereby obtaining an intermediate layer of mineral filler;[155] and still another in forcing the filler into the melted coating by means of an air-blast.[156]

Black Pigments.

These serve to exclude the actinic rays of the sun, which contribute largely to the decomposition of bituminous substances, and hence increase the weather-resistance of the coating. Opaque pigments are suitable for this purpose.

Colored Pigments.

For this purpose bright mineral pigments of various colors are mixed throughout special bituminous compositions exhibiting a brown color[157] when viewed on glass in a thin layer (0.001 to 0.002 in. thick), otherwise having a brown streak on porcelain (Test 6), e.g., gilsonite, fatty-acid pitch, asphaltic resin, montan wax, etc.; sometimes fluxed with fatty oils, wool-fat, etc.[158] The following schematic outline will indicate suitable mixtures for this purpose:

$$\left.\begin{matrix}\text{Gilsonite}\\ \text{Hard fatty-acid pitch}\\ \text{Montan wax}\end{matrix}\right\} \pm \left\{\begin{matrix}\text{Asphaltic resins}\\ \text{Medium fatty-acid pitch}\end{matrix}\right\} \pm \left\{\begin{matrix}\text{Soft fatty-acid pitch}\\ \text{Wool fat.}\end{matrix}\right.$$

Bituminous compositions having a black streak, or otherwise appearing opaque when viewed on glass in a thin layer, will not answer, for although they may initially show the color of the pigment, nevertheless this will disappear after a time turning a dull black, since *black* bituminous mixtures weather to intensely black substances, due doubtless to the liberation of free carbon by the actinic light rays.

Other mixtures that have been recommended for use in preparing colored coatings include: a mixture of fatty-acid pitch, asphaltic resin (i.e., "albino asphalt"), ground glass and colored pigments;[159] pine tar and colored pigment, which however lacks weatherproof properties;[160] mixtures containing rubber and mineral pigments, which also lack weatherproof properties;[161] etc.

Pigmented bituminous coatings are much more lasting than would result from the application of paints to the black bituminous coating, since most paints, and especially those composed of linseed oil, will soon disintegrate when applied over a plastic bituminous

base. It has been suggested, however, that if the bituminous base is heated to 300° C. with calcium oxide, zinc oxide, manganese dioxide, or lead oxide, either singly, or in various combinations, the coating may be painted with oil paints without danger of deterioration;[162] or if the saturant is composed of a mixture of rosin and dead oil;[163] or a mixture of fatty-acid pitch, rosin and linseed or paraffin oil;[164] or by first treating the bituminous coating with $CaOCl_2$, $K_2Cr_2O_7$, HNO_3, chlorine gas, nascent oxygen, or ozone;[165] or by affixing a layer of paper to the bituminous coating to which the paint is subsequently applied.[166]

Inorganic Surfacings. Three types of mineral surfacings are used, viz.: fine mineral particles, moderately coarse mineral granules and coarse mineral granules. Each will be considered separately.

(*A*) *Fine Mineral Particles.*

The fine mineral dust is sifted on the surface of the roofing usually while still hot, for the purpose of preventing the convolutions of the roll from sticking together after it is wound up, or otherwise packed for shipment. A certain proportion of it embeds itself in the surface coating, but most remains detached, so that it will either shake out of the roll, or wash off the roof when applied to the building. The fine dusting finish is therefore applied from a utilitarian standpoint rather than for decorative effects. The following materials have been used for the purpose:

(*a*) Uncolored Particles,[167] including ground talc, silica, infusorial earth, insoluble silicates,[168] slate flour, limestone,[169] Portland cement, mica dust and ground ashes.

(*b*) Colored Particles, including flake graphite,[170] mineral pigments,[171] aluminium powder or metallic bronzes,[172] iron filings,[173] metal flakes,[174] zonolite or vermiculite,[175] micaceous iron oxide,[176] etc.

(*B*) *Moderately Coarse Mineral Granules.*

These are embedded in either the top or bottom surface-coating for decorative effects. The mineral granules should be practically free from fines, and consist of uniformly sized granules, preferably angular. The following materials have been used:

(*a*) Uncolored Granules, including coarsely ground talc;[177] mica flakes;[178] sand;[179] rock or quartz;[180] marble or dolomite;[181]

crushed vitrified porcelain of a white color, obtained as waste in manufacturing bath-tubs, etc.

(*b*) Colored Granules, including the following types of products:

I. Naturally Colored Types: Natural slate in various colors (e.g., green, red, steel-blue, blue-black, black, grey, purple, etc.);[182] greenstone (altered diabase);[183] serpentine mixed with short-fibered asbestos;[184] river sand of yellow to brown color; sands of sundry colors.[185]

II. Ceramic Types (Vitrified Throughout):[186] Crushed bricks or tiles;[187] spangle, glass or smalt;[188] baked preformed particles in various colors;[189] crushed slag (e.g., iron, copper, gold or silver);[190] crushed slag (e.g., iron, copper or lead) fused with silica, or lime and sand;[191] crushed slag (e.g., copper or brass) fused with limestone.[192]

III. Ceramic Types (Vitrified Superficial Glaze): Glaze of borax and mineral oxide on mineral granules (e.g., pyrophyllite);[193] glaze of glass or "cullet" containing mineral oxides fused on mineral granules;[194] enamel fused on a porous mineral base;[195] ferrous sulfate and a fluxing agent (e.g., metallic borates, phosphates, etc.) fused on slag granules;[196] mineral oxide (e.g., CaO), phosphoric acid and mineral pigment set by heat or mineral granules.[197]

IV. Silicated Type: Slate or other mineral granules coated with sodium silicate and mineral pigments, rendered insoluble by heat treatment.[198]

V. Cement-coated Type: Mineral granules coated with Portland cement or Sorel cement mixed with mineral pigments.[199]

VI. Sulfurized Type: Mineral granules coated with melted sulfur mixed with mineral pigments.[200]

VII. Metallized Type: Metal granules;[201] mineral granules coated with metals electrolytically,[202] or by spraying.

VIII. Chemical Type: Inorganic pigments precipitated on mineral granules *without* thermo-treatment.[203]

IX. Thermo-chemical Type: Slate granules heated with sodium dichromate and ferrous sulfate or sulfuric acid to produce a brown color;[204] slate granules heated with sodium dichromate and copper sulfate to produce a reddish brown or chocolate color;[205] slate granules heated with sodium dichromate, copper sulfate and ferrous sulfate to produce a greyish-black color;[206] slate granules or sand heated with copper sulfate and ferrous sulfate to produce yellowish to purplish red colors;[207] particles of fire-clay or shale heated with ferrous sulfate or other metallic salts to produce reds;[208] particles of shale or slag heated with or without the addition of metallic salts;[209] mineral granules heated with a mixture of phosphoric acid and mineral pigments (e.g., metallic oxides).[210]

X. Painted Type: Sand or grit coated with a baked pigmented oil paint;[211] slag coated with a pigmented oil- or tar-paint;[212] rock granules coated with a pigmented nitro-cellulose lacquer;[213] granules coated with a metallic bronze paint or lacquer;[214] granules coated with a pigmented rubber solution and vulcanized.[215]

XI. Dyed Type: Porous granules (e.g., clay) impregnated with an organic dye.[216]

XII. Miscellaneous Types: Granules of hard asphalt mixed with colored mineral granules.[217]

(*C*) *Coarse Mineral Granules.*

Coarse granules are similarly embedded in the surface-coating for decorative effects. They may be either uncolored or colored, as follows:

(*a*) Uncolored Granules, including small pebbles or gravel;[218] crushed feldspar or granite;[219] granulated slag;[220] granulated coke;[221] granulated pumice;[222] or crushed sea-shells.[223]

(*b*) Colored Granules, such as coarse particles of slate.

Organic Surfacings. The materials in this group include wood flour or sawdust;[224] granulated cork;[225] granulated cornhusks, straw;[226] pellets of ground roofing waste;[227] granules of hard asphalt or asphaltites;[228] cordage or yarn;[229] brush-like filaments;[230] etc.

These are embedded in the surface coating while it is hot. The wood flour prevents the convolutions of the rolls from sticking together, and at the same time promotes the adhesion between the fabrics and the hot cementing compound in constructing built-up roofs or waterproofing membranes. Cork is used primarily for insulative effects.

SATURATED FELT

Saturating the Fabric. Felted or woven fabrics are impregnated with the bituminous saturating mixture by one of the following procedures:

1. By running the preformed web through a tank of the molten bituminous saturant. The impregnation may take place as follows:

(*a*) At atmospheric pressure in an open tank.[231]

(*b*) Under vacuum, using an open or closed tank provided with mechanism to operate continuously.[232]

(*c*) Under greater than atmospheric pressure in a closed tank.[233]

(*d*) By impinging the melted saturant under pressure through perforated rolls against the web of felt.[234]

2. By subjecting the freshly formed web of felt, just as it comes off the felt machine, and while it is still wet, to the highly heated saturant, for the dual purpose of expelling the moisture and impregnating the felt.[235]

3. By grinding or otherwise mixing fibrous substances with the saturant (either hot or cold), with or without the use of water, either as a mechanical mixture, or in the form of an aqueous dispersion in which the fibers act as the dispersing agent, and then rolling the mass into sheet form between calendar rolls, or else running the aqueous mixture over the felt machine.[236]

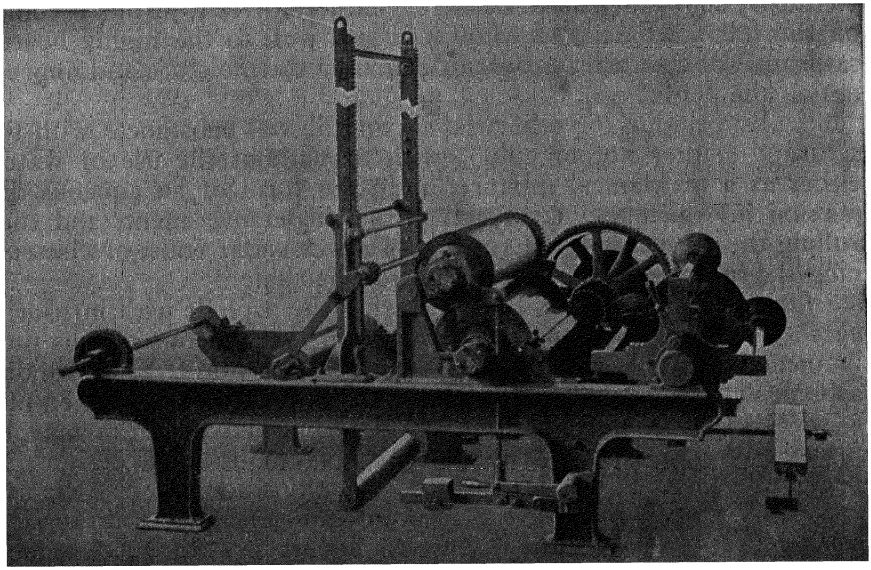
One example consists in first mixing the asphalt with infusorial earth and then dispersing the mixture in the aqueous fibrous pulp by means of a masticator;[237] another in mixing pulverized Trinidad asphalt with asbestos or other fibers and water which is formed into a sheet over the felt machine.[238] The finished products may be pressed or molded in various shapes, or embossed in designs, as previously described under "Molded Compositions."

4. By first converting the saturant into an aqueous dispersion, by means of any of the dispersing agents previously described, adding the emulsion to the aqueous suspension of fibers in the beaters, and then precipitating the bituminous matter on the fibers by adding slaked lime, alum, sugar of lead, sodium silicate, or the like, which serve to modify or precipitate the dispersing agent and thereby flocculate the bituminous matter. The sheet is then formed on the felt machine and dried in the usual manner.[239]

The method usually employed consists in treating the preformed web with the melted saturant at atmospheric pressure. The trade recognizes two types of saturated fabrics, viz.: those impregnated with tar products,[240] and those impregnated with asphaltic products.[241]

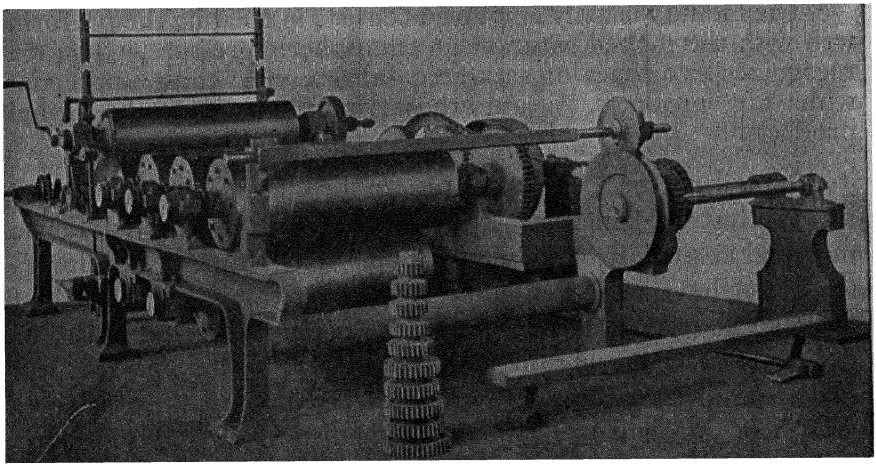
A modern form of apparatus for producing tar-saturated fabrics is illustrated in Fig. 145. The roll of unsaturated fabric is mounted at the left of the illustration. The frame or "gallows" in the center lowers the web into a steam-heated tank carrying the hot tar (not shown) by the means of the crank illustrated in the foreground. After leaving the bath, the saturated sheet passes around two steam-heated rolls (shown at the right of the frame work), and thence around the winding mechanism shown at the extreme right of the illustration.

A machine for saturating fabrics with asphalt is shown in Fig. 146. The roll of unsaturated fabric is supported at the rear of the illustration, whence it is lowered into the tank of the melted



Courtesy of The Moore & White Co.

FIG. 145.—Tar Saturator.



Courtesy of The Moore & White Co.

FIG. 146.—Asphalt Saturator.

asphalt (not shown), by the vertical frame operated by the crank shown at the left. After leaving the tank, the saturated sheet passes between two steam-heated press-rolls one above the other, located in front of the frame, and then around five steam-heated rolls mounted in two tiers, three on top and two below. These drive the excess of asphalt into the sheet, causing the saturated fabric to leave the machine perfectly "dry." It is then wound in large rolls on the automatic winder shown in the foreground.

The tanks in both the tar and asphalt saturators may be heated either by steam or direct fire. The former is ordinarily used because of the safety and ease of control. Tar products are usually maintained at 200–270° F., and asphaltic products at 225–400° F. In the types of machines illustrated, the fabric may be saturated at a speed of 330–350 lineal feet per minute. The weight of saturation absorbed by the fabric is controlled by the depth to which the web is immersed in the tar or asphalt, which in turn is regulated by raising or lowering the frame. The surface of the web in contact with the saturant varies from 25 to 100 lineal feet.

The following figures will give some idea of the percentages of saturating materials taken up by the fabric, based on its raw weight:

Felt will absorb an average of 125–200 per cent of soft coal-tar pitch, or 100–175 per cent by weight of asphalt.

Burlap will absorb an average of 90–125 per cent of soft coal-tar pitch, or 75–100 per cent by weight of asphalt.

The larger the percentage of any given saturant absorbed by the felted or woven fabric consistent with the formation of a "dry" surface, the more durably will the fibers be waterproofed and weather-proofed. If the felt is undersaturated, the finished sheet will be porous and absorb moisture in service. On the other hand, if the sheet is oversaturated, a part of the saturant will remain on the surface and cause the convolutions of the roll to stick together, but this may be obviated by surfacing the saturated sheet with fine sand or with powdered hard coal-tar pitch.[242] The best practice consists in running a sheet so that it will show "wet spots" as it leaves the bath of saturant, which, however, will become absorbed when it cools to atmospheric temperature. The absorption may be assisted by preventing the surface cooling too quickly, so that the

natural contraction of the saturant will have an opportunity of drawing in the "wet" spots by capillarity.

It has been suggested that the dry felt be saturated as it leaves the last dryer of the felt machine, while it is still warm, by making the saturator an integral part of the felt machine,[243] or by applying the melted saturant to the wet web of felt in between the dryers of the felt machine.[244]

The speed of saturation and the quantity of saturant taken up by the sheet may be increased by increasing the area of contact; [245] or by spraying the saturant on one side of the sheet; [246] or by spraying the saturant on both sides of the sheet; [247] or by contacting one surface of the sheet with the melted saturant; [248] or by flowing the melted saturant over both surfaces of the sheet as it passes around a vertical stack of steam-heated rollers; [249] or by passing the web of felt successively through two or more baths of the melted saturant maintained at different temperatures and cooling in between; [250] or by using corrugated rolls to apply the saturant; [251] or by forming a plurality of depressions on one or both faces of the sheet, which will become filled with saturant and upon cooling will be absorbed into the fabric; [252] or by applying pressure to the sheet while it is immersed in the saturant; [253] or by steaming the dry felt immediately prior to its saturation; [254] or by passing the saturated sheet through a heated absorption chamber; [255] or by conveying the saturated sheet vertically in between scrapers.[256] It has also been proposed to season the finished rolls of saturated fabric by heating them in a chamber to 158° F. or over.[257]

The web of fabric may be fed through the saturator without interruptions when splicing, by equipping the saturator with a festooning or looping device,[258] or by means of an automatic threading attachment,[259] or strips of gummed paper are recommended to splice together the ends of successive rolls,[260] or by sewing the ends together,[261] etc. A sheet-guiding device [262] has also been proposed; likewise the expedient of applying guide lines to the dry felt before saturating, by applying a mixture of bronze powder in sodium silicate, to facilitate laying same on the roof.[263] Foaming of the melted saturant may be inhibited by spraying the fresh saturant onto the surface of the body of saturant in the tank; [264] or by passing the freshly saturated sheet through a flame.[265]

In the United States, it is customary to market tarred felts in rolls of the dimensions and weights given in the following table:

	Pounds per 108 Sq. Ft.		Square Feet per Roll		Width, Ins.
	Usual	Occasional	Usual	Occasional	Usual
No. 1	26 and 27	24 and 25	216	250	32 and 36
No. 2	15 and 16	14 and 17	432 and 216	324	32 and 36
No. 3	13 and 14	11 and 12	432	.....	32 and 36

Tarred slaters' felt is marketed in 500 sq. ft. rolls, 36 in. wide, weighing 30 lb., and asphalt-saturated felts in rolls 32 or 36 in. wide weighing either 14 or 29 lb. per 100 sq. ft. and put up in multiples of a "square" (108 sq. ft.).

In Germany, the specifications for tarred roofing felt have been accurately standardized by the industry.[266]

Felts saturated with tar or asphaltic products are used for the following purposes:

(1) As an intermediate product in the manufacture of "prepared roofings" and composition shingles.
(2) In the manufacture of bituminized floor coverings.
(3) For constructing "built-up" roofs.
(4) For constructing waterproofing membranes.
(5) For bituminous expansion joints.

Saturated woven fabrics include the following groups of materials:

(1) Tarpaulins, used as covers ("paulins") for wagons, shock and hay covers, protecting machinery or merchandise on wharves, vessels, etc.; also covers for small portable structures. They consist of cotton canvas, duck, or drill, also hemp cloth, impregnated with bituminous compositions—usually of a dark color.

(2) Brattice cloth used for separating the passageways or shafts in mines to form air-ducts, or to confine the ventilation to prescribed routes. These consist of cotton fabric treated with dark-colored bituminous compositions, sometimes admixed with rubber.[267]

(3) Tents and awnings, which may consist of cotton canvas or duck treated generally with light-colored bituminous compositions.

(4) Waterproof membranes, which consist of canvas or burlap impregnated with coal-tar pitch or asphalt, and used in two or more layers, cemented together with melted coal-tar pitch or asphalt, respectively, in sub-soil waterproofing.

(5) Wrapping cloth, used to protect heavy machinery during transportation, composed of an open-mesh fabric impregnated with asphalt, or a mixture of asphalt and mineral oil.[268]

(6) Teredo-proof coverings used to protect wooden structures in tropical waters from attacks by marine borers (e.g., "teredo navalis"). This may be effectuated by coating the wood with melted asphalt and wrapping with the bituminized fabric, which is finally nailed in place.[269] Instead of using asphalt, the wood may be coated with a mixture of asphalt and mineral wax (e.g., zietrisikite);[270] or a mixture of asphalt with ground glass;[271] or with a mixture of asphalt, creosote oil, sulfur, mineral fillers and powdered copper.[272]

(7) Roof coverings for railway cars, which will be described later.

For exposure to the weather, mixtures of asphalt, petroleum or wool grease and lead oleate have given good results.[273] The addition of pigments to the waterproofing composition has been found to retard the deteriorating effects of the weather, due to their light-excluding properties.[274] The main problem, however, is to prevent the fabric from mildewing, especially when light-colored saturants are employed.[275] Paraffin wax in particular seems to promote the growth of mildew. The following substances have been found to retard or prevent the ravages of mildew: chrome alum and glue emulsified with asphalt;[276] an alkaline solution of trichlorphenol (2 : 4 : 6) used to treat the canvas prior to saturation;[277] the orange-colored resins obtained upon distilling coal-tar pitch immediately prior to coking, mixed with paraffin wax;[278] pine tar; sulfurized fatty-acid pitch;[279] cadmium oleate;[280] copper or zinc oleate, resinate, naphthenate, etc.;[281] a mixture of CuO, creosote oil, coal tar and wood tar;[282] a solution of copper resinate in water-gas tar oil; etc. An alternate procedure consists in treating the fabric with mineral salts prior to its impregnation with the bituminous substance.[283] Among the mineral salts used, may be included: sodium arsenate, mercuric chloride, zinc chloride, copper sulfate, aluminium sulfate, sodium fluoride, thorium fluoride;[284] sodium fluosilicate; sodium salicylanilide;[285] phenol and aniline derivatives; etc.

Table LXVII contains typical specifications for bituminized fabrics intended for built-up roofing and waterproofing work.

"Prepared roofings" are distinguished from "saturated fabrics" by being composed of: (1) Single-layered fabrics saturated and coated with bituminous compositions; (2) laminated (multiple-layered) bituminized fabrics having at least one of the layers saturated, the fabric being either unsurfaced, or surfaced on one side, or else surfaced on both sides with bituminous compositions. In the next succeeding section, we will consider prepared roofings composed of a *single* layer of fabric.

PREPARED ROLL-ROOFINGS

(A) Single-layered Prepared Roofings. The tarpaulin industry dates back at least to 1831.[286] In the early days, the fabric, either in the form of a hand-made "bat," or else in the form of a felt made continuously on a machine, was saturated cold with tar or asphalt paint, but this was soon followed by impregnating the fabric with the melted saturant which solidified upon cooling,[287] and in certain cases surfacing with sand. The next step in the development of the industry consisted in both saturating and coating a single web of fabric with bituminous mixtures, and surfacing with talc, sand, powdered limestone or gravel.[288]

Most of the prepared roofings manufactured to-day are made up of a single layer of "roofing felt" saturated with bituminous ingredients of relatively soft consistency, and surfaced on both sides with a harder bituminous composition. They are marketed in roll form, either 36 in. or 32 in. wide, measuring 108 sq. ft. in area, known commercially as a "square," which is sufficient to cover 100 sq. ft. of roof surface, allowing for 2-in. laps at the joints. Prepared roofings are marketed in four weights, known as "light," "medium," "heavy," and "extra heavy," weighing 35, 45, 55 and 65 lb. per square gross when finished with fine mineral surfacing, inclusive of nails and lap-cement.

The surface may be manufactured smooth, or it may have a "veined" appearance as illustrated in Fig. 147. In either event, it is customary to sprinkle a "dusting finish" of finely powdered mineral matter [289] on the surfaces to prevent the convolutions of the roll sticking together during transit or in storage. It is desirable to apply the dusting finish while both coatings are hot, so that as much as possible will embed itself in the surfaces and will neither

TABLE LXVII

Specifications for Bituminized Fabrics for Built-up Roofing and Waterproofing

Test No.	Description of Test	For Roofing	For Roofing and Waterproofing		For Flashings	For Waterproofing	
		Asbestos Felt	Rag-Felt		Rag-Felt	Woven Cotton	Woven Burlap
		Saturated with Asphalt	Saturated with Asphalt	Saturated with Coal-tar Pitch	Saturated with Asphalt	Saturated with Asphalt or Coal-tar Pitch	Saturated with Asphalt or Coal-tar Pitch
		A.S.T.M.[290]	A.S.T.M.[291] F.S.B.[292]	A.S.T.M.[293] F.S.B.[294]	F.S.B.[292]	A.S.T.M.[295] F.S.B.[296]	A.S.T.M.[297]
65	Width of roll, inches.....Range	36 or 32±½	36 or 32±½	36 or 32±½	36 or 32±½	30–38	30–38
65	Selvage edge, width, inches.....Max.	.....	.....	.....	.....	1/16 (*a*); ¼ (*b*)	3/16
65	Gross weight of roll as furnished, lb.....Range	40–80	40–80 (*a*); 50–80 (*b*)	40–80 (*a*); 45–80 (*b*)	50–80	35–80 (*a*)	35–80
65	Weight wrapping and packing per roll, lb.....Max.	½	½	½	½	2½	2½
65	Weight detached comminuted surfacing per 100 sq. ft., lb ..Max.	.....	1 (*b*)	.....	.....	1 (*a*)	1
65	Net weight material per 100 sq. ft., lb.....Aver	14±1	14±1	14±1	29±1.5		
65	Net weight material per sq. yd., oz.....Min.	.....	.....	.....	.....	11 (*a*); 11–15 (*b*)	20
68*a*	Pliability—shall not crack at 77° F., on mandrel, in.....	1/16	1/16	1/16	1/16	.....	
68*a*	Pliability—shall not crack at 32° F., on mandrel, mm.....	.....	.....	.....	.....	10 (*a*); 1/32 (*b*)	10
67	Strength at 70° F., with fiber grain, lb.....Min.	.....	30 (*b*)	30 (*b*)	40	50	70
67	Strength at 70° F., across fiber grain, lb .....Min.	.....	15 (*b*)	15 (*b*)	20	40	50
74	Moisture content, based on net weight, per cent.....Max.	.....	.....	2.5 (*a*)	2	1	1
71	Loss on heating at 221° F., 5 hrs., per cent.....Max.	.....	4	.....	4	4 (*a*) & 2 (*b*)	4
75	Saturant in moisture-free fabric, per cent.....Min.	50	140	140 (*a*)	150	200	200
16*b*	Extracted saturant:						
	Distillate from start to 210° C. (per cent by wt.).....Max.	.....	.....	2 (*a*)			
	Distillate from start to 235° C. (per cent by wt.).....Max.	.....	.....	5 (*a*)			
77	Weight per 100 sq. ft. of moisture-free fabric, lb .....Min.	9	5.2	5.2 (*a*)	10.4		
77	Weight per square yard of moisture-free fabric, oz.....Min.	.....	.....	.....	.....	3.5	7.5
78	Thickness of desaturated fabric, in.....Min.	.....	.....	0.025 (*b*)			
76	Ash in moisture-free desaturated fabric, per cent.....Max.	Min. 73 (*c*)	10	10	10	1	3
83	Fiber composition of fabric.....	.....	.....	.....	.....	Cotton 100 per cent (*d*)	Jute & manila 100 per cent.

Unless otherwise indicated, the above figures are common to both the A.S.T.M. and F.S.B. specifications.
(*a*) Included only in A.S.T.M. specifications. (*b*) Included only in F.S B. specifications.
(*c*) Presupposes a minimum of 85 per cent asbestos fibers carrying 14 per cent water of hydration (which is lost on ignition).
(*d*) The thread count both in direction of warp and filling shall be maximum 36 and minimum 18 per inch.

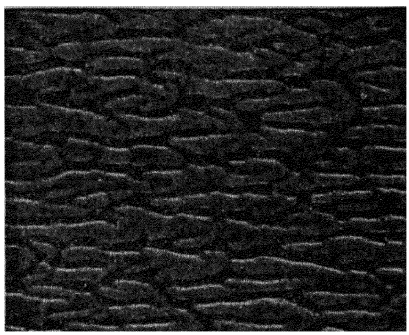


FIG. 147.—Prepared Roofing Finished in a Veined Surface.

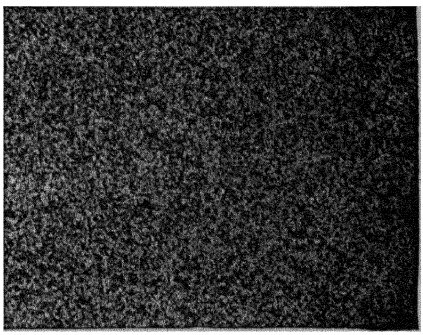


FIG. 148.—Prepared Roofing Finished with Coarse Talc.

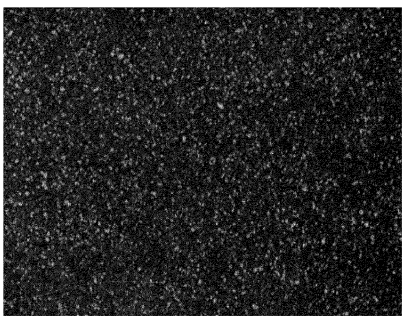


FIG. 149.—Prepared Roofing Finished with Fine Sand.

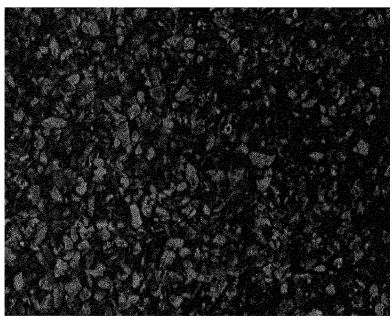


FIG. 150.—Prepared Roofing Finished with Crushed Slate.

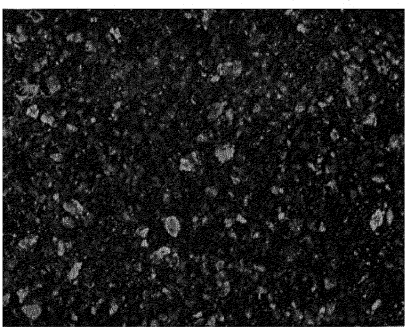


FIG. 151.—Prepared Roofing Finished with Crushed Feldspar.

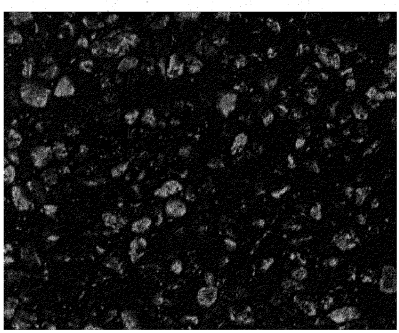


FIG. 152.—Prepared Roofing Finished with Quartz Pebbles.

blow away nor be washed off the roofing by the first rainstorm, after being laid on a building. Not more than 3 lb. per square is necessary for this purpose. It has also been suggested that the dusting-finish be applied as a suspension in water.[298]

In other cases, either one or both surface coatings may be adorned with moderately coarse particles of mineral matter, including coarsely ground talc, of which 9 to 12 lb. are applied per square to both sides of the sheet (Fig. 148); small rounded grains of sand, of which 10 to 15 lb. per square are used, including both sides of the roofing (Fig. 149); or angular particles of crushed slate (Fig. 150), of which 30 to 35 lb. per square are affixed to one side of the sheet. Crushed slate as supplied by the leading producers in the United States tests as follows on the Tyler type of sieves: Passing 8- and retained on 10-mesh sieve 4½ per cent, passing 10- and retained on 14-mesh sieve 34½ to 39 per cent, passing 14- and retained on 35-mesh sieve 55 to 59½ per cent. Another specification is as follows: Passing 8- and retained on 10-mesh sieve 2 per cent, passing 10- and retained on 14-mesh sieve 49 per cent, passing 14- and retained on 20-mesh sieve 25 per cent, passing 20- and retained on 28-mesh sieve 12 per cent, passing 28- and retained on 35-mesh sieve 6 per cent, passing 35- and retained on 48-mesh sieve 3 per cent, and passing 48-mesh sieve and retained on pan 3 per cent.

In other cases, coarse particles of mineral matter are embedded in the upper surface coating only, including crushed feldspar, of which 30–40 lb. are used per square (Fig. 151), or even large pebbles, of which 45–65 lb. are applied per square (Fig. 152).

The composition of typical prepared roofings is shown in Table LXVIII, and the specifications of single-layered roll-roofings as well as composition shingles are given in Table LXIX.

In manufacturing single-layered prepared roofings, the sheet of felted or woven fabric is first saturated by passing it through a bath of the melted bituminous saturation. The saturated sheet may then be coated immediately with a bituminous composition and while it is still hot, or else it may be wound into large rolls, allowed to cool and coated later. Both methods are used at present, the latter being known as the intermittent and the former as the continuous process. Each is claimed to have its particular advantages. The author is inclined to favor the continuous process, since the coating

is applied directly to the hot sheet, which enables the saturating compound on cooling and contracting to draw the hot and plastic surface coating into the sheet, forming a "key" between the coating and the fabric, and producing a *denser* sheet of roofing.

A modern type of high-speed machine for manufacturing roofing continuously is shown in Fig. 153.[299] The sheet travels from left to right, first over a splicing-looper for the dry felt,[300] then around a stack of steam-heated cylinders for preheating the felt,[301] then through a "saturator," [302] consisting of a deep tank heated by steam

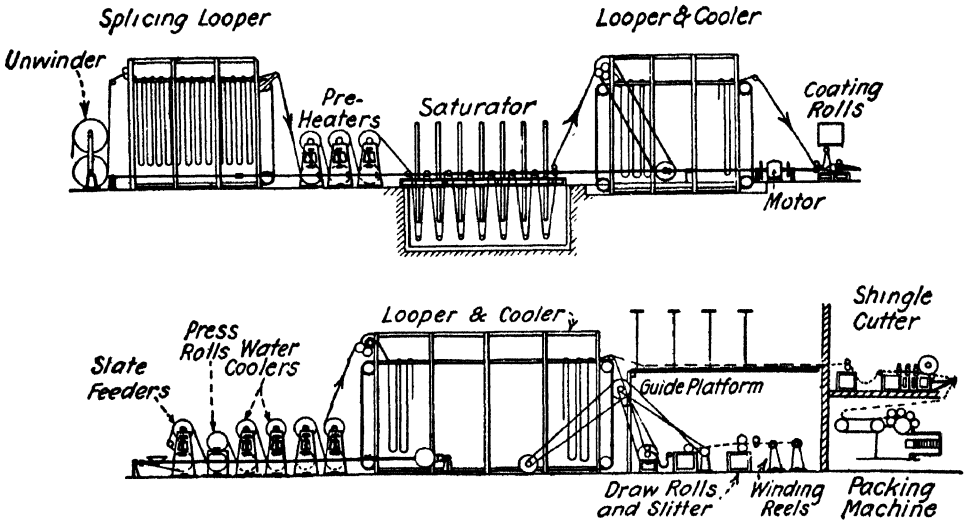


FIG. 153.—High-speed, Continuously Operating Machine for Manufacturing Prepared Roofing.

or direct fire, and then around a stationary air-cooled looping system.[303] The surface coatings are next applied to both sides of the sheet,[304] and while still hot, mineral particles are sifted on the upper surface from the so-called "slate feeder," [305] or they may be blown onto the coating by means of compressed air.[306] The sheet then travels around ten water-cooled drums, of which the first two serve as press-rolls to firmly embed the particles in the coating.[307] After this, the web is carried along by a movable looping device,[308] which serves to cool and store the finished sheet until it can be handled by the draw-rolls, automatic cutter for roll-roofings,[309] winder for roll-roofings,[310] or the shingle cutting and packing machine,[311] shown

TABLE LXIX

Test No.	Description of Test	Roll-Roofings and Cap-Sheet							Shingles	
		Fine Talc or Mica Surfacing		Coarse Talc Surfacing		Colored Granular Surfacing			Colored Granular Surfacing	
		Rag-Felt		Rag-Felt		Rag-Felt		Asbestos Felt	Rag-Felt	
		Med'm Wt.	Heavy Wt.	Heavy Wt.	Extra Heavy Wt.	Stand. Wt.	Cap-Sheet	Stand. Wt.	Stand. Wt.	Heavy Wt.
		A.S.T.M.[312] F.S.B.[313] Und. Lab.[314]	A.S.T.M.[312] F.S.B.[313] Und. Lab.[314]	A.S.T.M.[315] Und. Lab.[314]	A.S.T.M.[315] Und. Lab.[314]	A.S.T.M.[316] F.S.B.[317] Und. Lan.[314]	A.S.T.M.[318]	F.S.B.[319]	A.S.T.M.[320] F.S.B.[317] Und. Lab.[314]	A.S.T.M.[320] Und. Lab.[314]
65	Width of roll-roofings, in.....Range	36 or 32±¼	36 or 32±¼	36 or 32±¼	36 or 32±¼	36 or 32±¼	36 or 32±¼	36 or 32±¼	Sufficient to protect 100 sq. ft. of roof area (*g*)	
65	Area of roll-roofings, sq. ft.....Min.	108 or 216(*ab*)	108 or 216(*ab*)	108 or 216(*a*)	108 or 216 (*a*)	2 in. edge: 108 3 in. edge: 111	36 in.: 106.8±5% 32 in.: 107.3±5%	108	.....	
65	Size and form of shingles..	.....	.....	.....	.....	.....	.....	.....	As may be specified (*b*), or as may be agreed upon (*a*), or as approved (*c*)	
65	Gross weight per 108 sq. ft., lb....Aver.	45 (*b*) 55 (*a*)	55 (*b*) 65 (*a*)	55 (*a*)	65 (*a*)	90 (*a*)	.....	.....	.....	.....
65	Wrapping, nails and cement per 108 sq. ft., lb.....Max.	3 (*b*)	3 (*b*)	.....	.....	5 (*b*)	1	.....	.....	.....
65	Net weight of material per 108 sq. ft., lb.....Aver.	42 (*c*) 52 (*a*)	62 (*a*)	42 (*c*) 52 (*a*)	62 (*a*)	No edge: 82 (*c*), 83 (*a*) 2–3 in. edge: 81 (*a*)	55	.....	86 (*a*)	102 (*a*)
	Net weight of material per 108 sq. ft., lb.....Min.	40 (*bc*) 50 (*a*)	50 (*b*) 60 (*a*)	40 (*c*) 50 (*a*)	60 (*a*)	80 (*abc*)	52	80	83 (*ab*)	97 (*a*)
68*a*	Pliability test at 77° F. (*d*): Amount of cracking when bent in 2 seconds:									
	90° over a rounded edge of a radius.....½-in.	None (*a*)	None (*a*)	None (*a*)	None (*a*)	.....	.....	.....	.....	.....
	¾-in.	None (*b*)	None (*b*)	.....	.....	None (*ab*)	None	No requirement	No requirement	No requirement
	180° over a mandrel of a diameter.....0.4-in.	None (*c*)	None (*c*)	None (*c*)	None (*c*)	.....	.....			
	1.0-in.	.....	.....	.....	.....	None (*c*)	.....			

71	Loss and behavior (*e*) on heating for 2 hours at 176° F., per cent.....Max.	1.5	1.5	1.5	1.5	1.5 (*f*)	1.5 (*f*)	1.5 (*f*)	1.5 (*f*)	1.5 (*f*)
17*a*	Flash-point of saturant and coatings, ° F. .....Min.	410 (*c*)	410 (*c*)	410 (*c*)	410 (*c*)	410 (*c*)	.....	.....	410 (*c*)	410 (*c*)
76	Asphalt-saturant (sol. CS_2) in moisture-free felt { per cent.....Min.	{ 150 (*bc*) 160 (*a*)	150 (*c*) 160 (*ab*)	150 (*c*) 160 (*a*)	150 (*c*) 160 (*a*)	150 (*c*) 160 (*ab*)	} 150	60	{ 165 (*c*) 175 (*ab*)	165 (*c*) 175 (*a*)
	{ per 108 sq. ft., lb., Min.	17.3 (*a*)	21.6 (*a*)	17 3 (*a*)	21 6 (*a*)	17 3 (*a*)	.....	.....	18 9 (*a*)	27.5 (*a*)
76	Asphalt soluble in carbon disulfide, in.: Both surface coatings per 108 sq. ft., lb.....Range	15–20 (*a*)	15–20 (*a*)	.....	.....	.....	.....	.....	.....	.....
	Upper coating per 108 sq. ft., lb.Range	.....	.....	8–15 (*a*)	8–15 (*a*)	12–20 (*a*)	.....	.....	12–20 (*a*)	12–20 (*a*)
	Under coating per 108 sq. ft., lb.Range	.....	.....	Min. 2 (*a*)	Min. 2 (*a*)	2–5 (*a*)	.....	.....	2–5 (*a*)	2–5 (*a*)
76	Mineral-surfacings per 108 sq. ft., lb.Max.	2 (*ci*)	2 (*ci*)	2 (*ci*)	2 (*ci*)	35 (*ci*)	.....	.....	35 (*ci*)	35 (*ci*)
76	Mineral-matter pass ng No. 6 but retained on No. 100 sieve, per 108 sq. ft., lb.....Range	.....	.....	.....	.....	25–40 (*a*)	.....	.....	25–40 (*a*)	25–40 (*a*)
76	Mineral-matter passing No. 14 but retained on. No. 100 sieve, per 108 sq. ft., lb.....Range	.....	.....	3–12 (*a*)	3–17 (*a*)	.....	.....	.....	.....	.....
76	Mineral-matter retained on No. 100 sieve, per 108 sq. ft., lb.....	4–8 (*a*)	4–8 (*a*)	4–10 (*a*)	4–10 (*a*)	4–10 (*a*)	.....	.....	4–10 (*a*)	4–10 (*a*)
76	Total mineral-surfacings and mineral-matter in coatings, per 108 sq. ft., lb.....Max.	.....	.....	.....	.....	.....	45	35	.....	.....
	Total mineral ash in roofing per 108 sq. ft., lb..... Max.	9 (*b*)	9 (*b*)	.....	.....	.....	.....	.....	.....	.....
77	Moisture-free felt per 108 sq. ft., lb., Min.	10.8 (*ab*)	13.5 (*ab*)	10.8 (*a*)	13.5 (*a*)	10 8 (*ab*)	10.8	18.0	10.8 (*ab*)	15.7 (*a*)
77	Moisture-free felt, "number," lb. per 480 sq. ft.....Min.	48 (*c*)	.....	48 (*c*)	.....	48 (*c*)	48	.....	48 (*c*)	70 (*c*)
78	Moisture-free felt, thickness, in....Min.	0 048 (*b*)	0.060 (*b*)	.....	.....	0 050 (*b*)	.....	0 040	0.050 (*b*)	.....
76	Moisture-free felt, ash, per cent...Max.	10 (*c*)	.....	10 (*c*)	.....	10 (*c*)	12	Min. 70 (*h*)	10 (*c*)	10 (*c*)
76	Total asphalt (sol. CS_2) and organic fibers in roofing per 108 sq. ft., lb., Min.	.....	.....	.....	.....	45 (*b*)	.....	.....	45 (*b*)	.....

(*a*) Included only in A.S.T.M specifications.

(*b*) Included only in F.S.B. specifications.

(*c*) Included only in Und. Lab. specifications.

(*d*) Behavior of at least 8 out of a total of 10 strips tested.

(*e*) There shall be no flowing, sagging, blistering, or absorption of the surface-coatings; nor shall the granular surfacing become loosened or detached.

(*f*) Upon being tested in a vertical position, granular surfacings shall not slide more than 1/16 in.

(*g*) Includes individual and strip-shingles, which shall be of a form, shape and size that there shall be at least one thickness of material at all points over the roof-deck when laid, and so that no portion of the roof-deck shall be exposed upon cutting away the unsecured exposed portion of the shingles along a line through the nails securing them to the roof-deck.

(*h*) Asbestos-felt shall contain at least 85 per cent by weight of asbestos fibers, and the remainder may consist of hair, cotton-rags or linters, starch, or other suitable binding material.

(*i*) Should the weight exceed the figure stipulated, then the finished weight per 108 sq. ft. shall be increased by a corresponding amount.

TABLE

COMPOSITION OF

	Fine Talc Surfacing													
	Unfilled Coatings								Filled Coatings					
	Light		Medium*		Heavy*		Extra Heavy*		Light		Medium		Heavy*	
	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent
Mineral Matter:														
Detached surfacing—top and bottom	1.5	4 7	1.8	4 3	2.1	4 0	2.4	3.8	1.5	4 7	1.8	4 3	2.1	4.0
Fine talc, embedded in top coating	0 3	1 0	0.4	1.0	0.5	1 0	0.6	1 0	0.3	1 0	0.4	1.0	0.5	1.0
Fine talc embedded in bottom coating	0 2	0.6	0 3	0 7	0.4	0 8	0 5	0.8	0.2	0.6	0.3	0.7	0.4	0.8
Coarse talc embedded in top coating	.	.	..	...	...	.	.	...	..	..	..	...	..	...
Coarse talc embedded in bottom coating	...			...	.	.		.	..	...	...	..	...	....
Sand embedded in top coating	...			.			.	..	...	...	...	...	...	....
Sand embedded in bottom coating	...		.			..	.	.	..	..	...	...	...	....
Colored granules embedded in top coating	....	..	..	...	...	..	....	...	..	....	..	....	....	....
Filler admixed with top coating	...		...	...	.	.	...	..	4.5	14 1	5.1	12.1	5.4	10:4
Filler admixed with bottom coating	...	...	.	...	.	..	...	...	3.0	9 3	3.4	8.1	3.6	6.9
Filler admixed with cementing layer	..	.	.	..	..	.	...	..	...	..	..	...	....	....
Bituminous Matter:														
Contained in top coating	5 6	17.5	8.0	19.0	9.6	18.5	12.4	20.0	6.7	20.9	7.6	18.1	8.1	15.6
Contained in bottom coating	3 8	11 9	5.3	12.6	6.4	12.3	8.3	13.4	4.5	14.1	5.0	11 9	5.4	10.4
Contained in cementing layer	.	..	..	..	..	..	..	..	....	..	...	.	...	...
Saturating the fibrous matter	12.0	37 4	15.4	36 7	19.5	37.5	22.0	35.5	6.6	20.6	10 8	25.7	15.7	30.1
Fibrous Matter:														
Dry felt—one or more layers	8.6	26 9	10 8	25.7	13.5	25.9	15.8	25.5	4.7	14.7	7.6	18.1	10.8	20.8
Dry burlap	...	.	..	..	..	...	...	...	...	. .	.	...	..	...
Total per square (108 sq. ft.)	32	100	42	100	52	100	62	100	32	100	42	100	52	100
Fixtures:														
Nails, including container	1 6	...	1 6	...	1.6	....	1 6	..	1.6	.	1.6	....	1.6	...
Can of lap cement (1 pint)	1 4	..	1 1	.	1.4	...	1.4	..	1.4	...	1.4	...	1.4	...
Total weight per square (108 sq. ft.)	35	..	45	...	55	....	65	..	35	....	45	. .	55	....

* Approved by Underwriters' Laboratories, Inc. of

to the extreme right of the illustration. The cooling of the sheet may be facilitated by impinging jets of air, or a water-spray against it.[321]

One method consists in saturating and coating a sheet of double width and then slitting it longitudinally into two sheets of the desired width.[322] Another consists is passing two sheets through the machine, one above the other, to double the output.[323] Still another variation consists in corrugating the saturated web longitudinally

LXVIII

TYPICAL ROLL-ROOFINGS

Coarse Talc Surfacing						Sanded Both Sides						Colored Granules One Side				Double Layered		Burlap Center, Sanded	
Unfilled Coatings						Unfilled Coatings						Filled Coating							
Light		Medium*		Heavy*		Light		Medium		Heavy*		Medium*		Heavy*		Medium		Extra Heavy	
Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent	Lb.	Per Cent
....	....	....	....	.	...	..	...	..	.	...	...	0.7	0.8	0.7	0 7	1.6	3.1		
....	.....	....	.....	. .	...	...	...	...	...	...	.....	..	.	....	.	0.4	0.8		
....	.....	....	. .	.	...	...	....	....	...	....	.....	0.3	0 4	0 3	0.3	0.4	0.8		
4.5	10.7	5.0	9.6	5.5	8.9														
4.5	10.7	5 0	9.6	5.5	8 9														
....	.....	....	.....	....	....	4.75	14.8	5.5	13.1	6 0	11.55	....	..	.. .	...	....	....	7.5	10.4
....	...	..	....	.	....	4.75	14 8	5.5	13.1	6.0	11.55	....	....	.....	....	....	....	7.5	10 4
....	.....	...	.....	....	....	..	....	.	...	...	...	30.0	36.6	35.0	33.3				
...	.....	...	.....	...	...	...	...	...	...	..	...	4.0	4.9	4.0	3.8	2.1	4.0		
....	.....	....	.....	...	..	..	....	...	..	...	..	..	. .	.. .	...	2.1	4.0		
....	...	.	...	...	..	...	...	..	.	..	..	.	.	....	..	2.4	4.6		
7.4	17.6	9.5	18 3	10.8	17 4	6.7	20 9	7 6	18 1	8.1	15.6	17.0	20 7	18.65	17.8	4.2	8.1	5.8	8.1
5.0	11.9	6.3	12.1	7.2	11.6	4.5	14.1	5.0	11.9	5 4	10 4	3 0	3.6	3.0	2.8	4.2	8.1	5.8	8.1
....	.....	..	....	..	....	. .	....	..	..	.	..		..	...	..	4.8	9.3	10.5	14.5
12.0	28.6	15 4	29.6	19.5	31.4	6 6	20.6	10 8	25.7	15.7	30 1	16 2	19.8	27.6	26.3	8 1	15.5	8.1	11.2
																8.1	15.5	8.1	11.2
8.6	20.5	10.8	20.8	13.5	21.8	4.7	14 7	7 6	18 1	10 8	20 8	10 8	13 2	15.75	15.0	6.8	13.1	6.8	9.5
																6 8	13.1	6.8	9.5
...	....	...	....	..	.	.	..	. .	.	.	. .	..	...	...	....	...	...	5.1	7.1
42	100	52	100	62	100	32	100	42	100	52	100	82	100	105	100	52	100	72	100
1.6	....	1.6	....	1.6	.....	1 6	...	1 6	.	1.6	. .	1 6	...	1.6	....	1.6	...	1.6	....
1.4	.....	1.4	.....	1.4	.....	1.4	....	1.4	..	1.4	..	1.4	...	1.4	....	1.4	...	1.4	....
45	....	55	....	65	.....	35	. .	45	....	55	.....	85	.. .	108	....	55	...	75	...

Chicago and complying with their specifications.

before the coatings are applied.[324] A machine in which the various parts are superimposed above one another vertically in several floors has been described;[325] also a machine which automatically lifts the web of felt out of the saturator when the operation is stopped, to prevent the felt from being scorched.[326] A device has also been proposed which prevents the web of felt sliding or shifting from side to side during its progress through the machine.[327] For manufacturing smooth coatings, the use of electrically heated

smoothing rolls has been patented,[328] likewise the procedure of applying an endless band of paper, which is subsequently removed.[329] Defectively surfaced shingles may be resurfaced by first reheating, applying additional granules and finally subjecting to pressure.[330] Other expedients consist in roughening the coating with an air-blast before applying the granules;[331] applying the coating in two layers, the first containing an admixture of fibrous mineral or vegetable matter;[332] applying two successive coatings with a layer of granular mineral matter in between;[333] embossing the surface-coating in the form of intersecting ribs or the like to impart a distinctive appearance;[334] etc.

Mineral-surfaced roll-roofings are provided with a 2-in. marginal edge to facilitate forming the lap when laid.[335] This edge may be formed either by applying a strip of thin paper to the coating before the granules are affixed; or by embedding strips of paper or metal foil in the weather coating;[336] or by surfacing the edge of the sheet with fine-grained mineral matter of a contrasting color;[337] or by applying a coating of asphalt over the granules along the edge;[338] or by depressing the granules along the marginal edge;[339] or by chilling the hot coating along the edge so as to prevent the granules from adhering to this portion of the sheet;[340] or by omitting the granules from the marginal edge;[341] or by scraping off the hot coating along the edge;[342] or by forming a rib along the edge as a result of creasing same;[343] etc.

To render the surface of asphalt-coated roofings non-adhesive, it is proposed to coat the asphalt with a thin layer of wax,[344] or with a solution of cellulose nitrate, cellulose acetate, or viscose,[345] with or without the incorporation of a colored pigment or a metallic bronze; or with milk of lime;[346] or with a mixture of rubber, chlorinated-diphenyl and fillers;[347] or with a solution of alginate, soap, glue and glycerin.[348] To improve the color of the granular surfacings, and prevent their being affected by the oily matters in the underlying coating, a method has been patented consisting in the application of an aqueous solution of sodium silicate[349] or glue or soap bark,[350] likewise an aqueous solution of dextrine.[351] Similar results may be obtained by moistening the granules with an asphalt solvent and thus improve their adhesion;[352] or by mixing 3 to 4 per cent of powdered rosin with the granules;[353] or by treating

the granules with a solution of rosin in a volatile solvent.[354] To obviate blistering of the coating, it has been suggested that the saturated felt be dusted with zinc or calcium stearate before the asphalt coating or mineral granules are applied.[355] To prevent roll-roofings from wrinkling when laid on the roof, it has been proposed that the fabricated sheet be run through sets of corrugated rolls, so as to undulate the sheet transversely and longitudinally;[356] or by passing the freshly saturated sheet over a tank of boiling water.[357]

Very often prepared roofings surfaced with finely powdered mineral matter, such as talc or limestone, turn yellow on aging indoors. This discoloration is due to the use of:

(1) Bituminous coating or saturating mixtures containing a large proportion of volatile matter.
(2) Coating compositions which do not blend thoroughly.
(3) Coatings containing a large proportion of "greasy" or oily constituents which impart a dull surface to the specimen on aging indoors one week (Test 3).

This yellow discoloration not only detracts from the appearance of the roofing and interferes with its sale, but indicates that improper mixtures have been used in its manufacture.

The function of the coating is to seal in the saturation, protect it from oxidation and volatilization, and exclude moisture from the fabric fibers. The coating should weather away gradually and *uniformly*. As soon as the coating disappears through weathering or attrition of the elements, the fabric will commence to rot and weaken, until it tears during the first heavy wind, and this will terminate the life of the roofing.

A properly laid prepared roofing will no longer fulfil its function as a result of:

(1) Local perforations due to the coating compound failing in spots, the seams opening up on account of the lap-cement being faulty, hard lumps or particles in the felt, rusting away of the nails, the bituminous matter being extracted from the felt by rosin or turpentine in the roof boards, the fabric drying out or otherwise becoming brittle and cracking, the roof being subjected to undue external violence such as a hailstorm, rough walking upon it, dragging heavy articles over it, objects falling upon it, etc.
(2) The fabric weakening to such an extent that it is torn and

rendered unserviceable by the first heavy windstorm. As long as the weather coating remains intact, this will not occur, but just as soon as it wears thin or disappears, moisture will find its way into the fabric and rot its fibers, so that the roof is soon doomed unless it is repainted.

(3) Trouble caused by the underlying roof boards, due to shrinkage, cracking, splitting or rotting, which would subject the roofing to excessive strains, and either rip it from the seams or tear the sheets.

Most manufacturers recommend that prepared roofings be coated from time to time with a bituminous lacquer to renew the weather-coating as it wears away. This procedure is applicable to smooth-surfaced roofings, or ones surfaced with very fine sand or grit. Slate-surfaced roofings or roofings coated with gravel or crushed rock are not amenable to painting, due to the interference of the coarse mineral particles. With proper care and repainting at intervals, a properly manufactured single-layered heavy-weight smooth-surfaced prepared roofing will remain serviceable for twenty years. Some manufacturers guarantee light-weight roofing to last five years without painting, medium weight from five to ten years with one painting, and the heavy weight from ten to twenty years with two or more paintings. As in other industries, it is generally true that the smaller and less responsible the manufacturer, the more extravagant the guarantee offered. In recent years, guarantees have been very much overdone by certain prepared roofing manufacturers, and as a result the public has grown wary, but is fortunately beginning to pay more attention to the manufacturer's reputation and to the record of the particular brand of roofing, which after all afford much better assurance than a "guarantee." A method of identifying prepared roofings consists in embedding a plurality of spaced colored threads in the undercoating;[358] another consists in impressing an identifying mark on the undercoating;[359] or by embedding a plurality of spaced threads in the raw felt;[360] or by embedding strips of metal bearing identification marks in the raw felt;[361] or by printing the surface of the dry felt with an insoluble dye;[362] or by punching holes of predetermined spacing along the edges of the dry felt.[363]

A single sheet of *woven* fabric such as duck, coated on one side with a bituminous mixture dusted with powdered talc or mica, is

largely used at the present time for covering boat decks or porch roofs where subjected to foot traffic. The material is laid with the bituminous side down, whereupon the unsurfaced duck is painted with several coats of a colored linseed-oil pigment paint.[364] A single sheet of woven fabric saturated with a soft bituminous composition, coated on both sides with a mixture of harder consistency and surfaced with powdered talc or mica, is being used largely for roofing railroad passenger cars and locomotive cabs. This is manufactured from "numbered" ducks weighing 11.46 to 14.73 oz. per sq. yd., the finished weights being 2.15 and 2.30 lb. per sq. yd., respectively.

In Germany, a hemp fabric is impregnated with a moderate quantity of fatty-acid pitch, which is thereupon congealed or solidified by heating in air, and the surface of the sheet coated with a bodied linseed-oil-pigment composition and allowed to dry. The congealed fatty-acid pitch impregnation does not injuriously affect the oil coating upon exposure to the elements.[365]

(B) Laminated Roofings. These consist of two or more layers of bituminized felted or woven fabrics, sometimes in combination wire mesh or sheet metals, combined in innumerable ways. The principal combinations include the following:

(1) Two or more layers of bituminized felted fabrics cemented together with a bituminous adhesive,[366] including:

(*a*) Tarred rag-felts cemented together with coal-tar pitch.

(*b*) Asphalt-saturated rag-felts cemented together with asphalt.

(*c*) Asphalt-saturated asbestos felt cemented together with asphalt and in some instances surfaced with a sheet of unsaturated asbestos felt. An alternative procedure consists in cementing together two or more layers of unsaturated asbestos felt with relatively thick layers of asphalt.[367]

(*d*) Combinations of asphalt-saturated rag-felt with asbestos felts,[468] and sometimes with woven fabrics.

(2) Combinations of felted fabrics with woven fabrics,[369] including:

(*a*) Burlap embedded in an asphaltic adhesive between two layers of asphalt-saturated rag-felt and surfaced top and bottom with an asphaltic composition.

(*b*) Untreated cotton duck fastened with an asphaltic adhesive to an asphalt-saturated and coated felt. This is intended for use as a form of decking and porch covering, and to have its upper surface painted with a pigmented oil-point, along the lines previously described.[370]

(3) Combinations of asphalt-saturated felted or woven fabrics with wire mesh.[371]

(4) Combinations of asphalt-saturated rag- or asbestos-felt, or of woven fabrics with a core of sheet metal (e.g., lead, tinplate, or steel); [372] or surfaced with sheet metal, which may be either flat or corrugated, or may have metal strips cemented in between folds to reinforce same.[373]

(5) Asphalt-saturated rag-felt surfaced with a thin sheet of zinc or aluminium foil.[374]

(6) Combinations of asphalt-saturated and coated felt with a thin layer of ply-wood.[375]

Flexible laminated prepared roofings may be manufactured either by the continuous or the intermittent process. If the former is used, the individual layers are first impregnated in a tar or asphalt saturator, then wound in rolls of large diameter known as "jumbo" rolls, and finally joined in the desired number of layers and sequence on a form of machine illustrated in Fig. 154, designed to assemble not exceeding five layers. If made on a continuous-process machine, as many saturators must be provided as there are layers to be impregnated. The layers may be cemented together with melted asphalt, or with an aqueous dispersion of asphalt.[376]

Since a woven fabric (burlap or cotton ducking) absorbs a smaller percentage of bituminous saturation than a felted fabric, the former when saturated is far less weather-resisting . It is preferable, therefore, to use the woven sheet for *backing* a laminated roofing, and thus protect it from the direct action of the weather as much as possible, and where it will at the same time fulfil its function of adding to the strength of the finished product.

It is claimed that a roofing composed of a layer of rag-felt saturated and coated with bituminous matter, to which is cemented a thin sheet of paper, will successfully retain a coat of oil-plant on the upper uncoated side.[377] Similarly, a covering has been proposed

for withstanding the action of oil vapors on petroleum storage tanks, consisting of a layer of asphalt-saturated and coated felt, cemented with coal-tar pitch to a layer of paper, which in turn is surfaced on the underside with coal-tar pitch;[378] or by combining roll-roofing with a layer of paper impregnated with sodium silicate.[379]

(C) Decorative Roll-roofings. Numerous devices have been proposed for the purpose of ornamenting the surface of granular-surfaced roll-roofings so as to break up the continuity of the surface, and cause it to simulate the appearance of tiles or shingles, or otherwise to assume the form of patterns. The following expedients have been used:

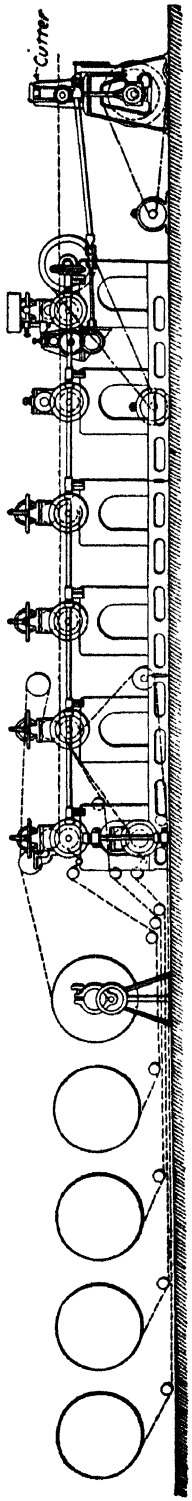
(1) Embedding in the asphalt coating, granules of two or more colors, either mixed together, or in mottled areas, or in striped, mosaic or other patterns.[380]

(2) Depositing the colored granules through a stencil or series of belts in patterns.[381]

(3) Affixing to the asphalt coating a non-adhesive material in stripes or patterns, and then embedding granules of two or more colors in the intervening areas.[382]

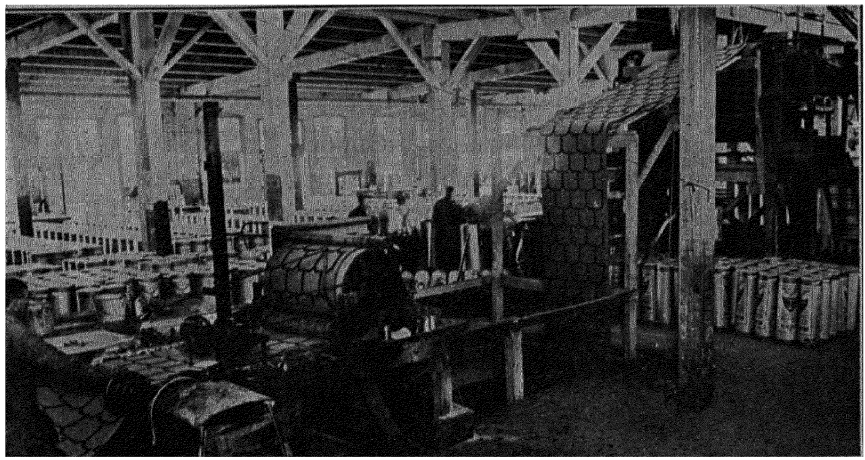
(4) Depressing or embossing or partly removing the colored mineral surfacing so as to expose the underlying asphalt coating in the form of patterns,[383] over which granules of a different color may be embedded in the depressed areas.[384]

(5) Applying liquid paints or lacquers in patterns over the colored mineral granules.[385] The paint may either be uncolored, or else a bituminous paint in the form of a clay dispersion of fatty-acid pitch or wax tailings mixed with a colored pigment, to which colored mineral granules may be affixed in stripes or designs.[386]



Courtesy of Guyton & Cumfer Mfg. Co.

FIG. 154.—Machine for Assembling Multiple-layered Bituminized Fabrics.



Courtesy Bird & Son.

FIG. 155.—Machine for Making Decorative Roll-roofing.

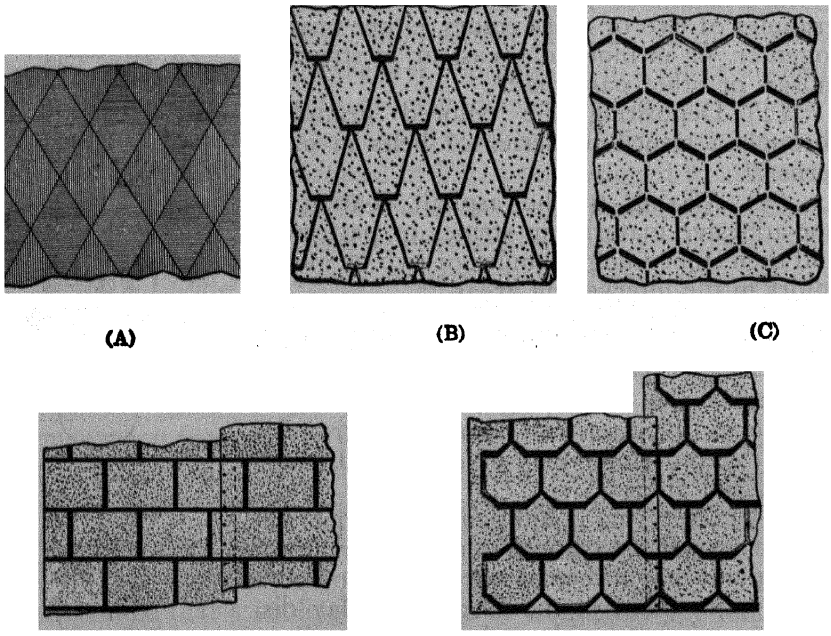


FIG. 156.—Roll-roofings Finished in an Ornamental Surface.

(6) Applying liquid bituminous paint over the colored mineral granules and then embedding differently colored granules therein.[387]

(7) Applying melted asphalt (in some cases admixed with a colored pigment) in patterns over the colored granular surfacing.[388]

(8) Applying melted asphalt in patterns over the colored granular surfacing and then embedding different colored granules therein.[389]

(9) Creasing, or folding, or plaiting the roofing in the form of patterns.[390]

(10) Embossing the sheet of roofing in the form of tiles and filling the depressions in the under side with a composition of matter.[391]

(11) Cementing triangular or circular patches of roofing to the underside of the sheet, so as to throw a pattern into relief.[392]

(12) Cementing subdivisions cut from a roofing sheet to the upper surface, in the form of patterns.[393]

These decorative sheets have become quite popular in recent years. They are less expensive than composition shingles in view of the smaller amount of material required to cover 100 sq. ft. of

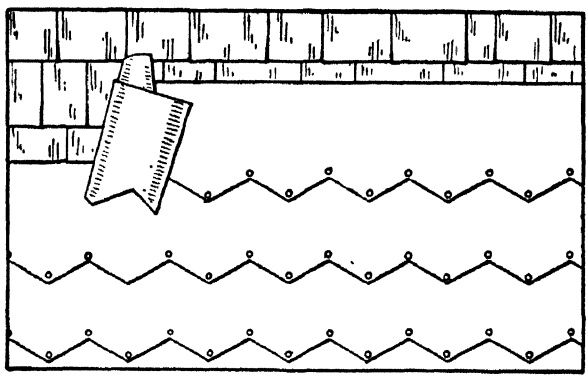


FIG. 157.—Diamond-point Roll-roofing.

roof surface, and are accordingly used where the higher price of the shingles cannot be afforded. Fig. 155 illustrates a machine for manufacturing so-called "print roofing," in which the design is formed by applying melted asphalt over the colored mineral granules as described in section 8 above. Fig. 156 shows a few of the popular designs on the market.

(D) Scalloped and Serrated Roll-roofings. These represent a further development in the art to produce decorative effects in imitation of wooden or slate shingles or tiles, which is brought

about by cutting the roofing in long strips adapted to be laid in overlapping courses. One edge of the sheet is cut in a scalloped or serrated edge which is exposed to the weather and nailed in place.[394] A popular form has diamond-shaped serrations as illustrated in Fig. 157. Another modification, known as "diamond print-roofing," has black stripes printed over the surface adjacent to the serrated edge.[395]

ASPHALT SHINGLES

These are cut in relatively small units from roofing surfaced with colored mineral granules, and are intended to be laid in overlapping courses in much the same manner as wooden shingles or slates. Prepared-roofing shingles are cut in a multitude of patterns, and are finished in many different ways, as will now be described.

General Features

(1) *Features Pertaining to Thickness*: Shingles may be manufactured of uniform thickness with cut edges exposing the central layer of felt; or with "sealed edges";[396] or the lower edge may be cut in a finely serrated (i.e., ragged) form to give an illusion of greater thickness;[397] or the lower edge may be cut in a ragged form and charred to accentuate the thickness.[398]

Shingles may also be manufactured in a tapered form with a thicker butt end, which may be accomplished in any of the following ways: applying the asphalt coating in a tapered layer;[399] applying two or three uniformly thick coatings with mineral surfacings to the lower portion of the shingle;[400] applying two or three coatings to the lower portion of the shingle with pellets of roofing embedded therein;[401] double coating and surfacing the lower portions of opposite sides of the shingle;[402] applying a double coating along the lower edge of the shingle;[403] forming a laminated shingle composed of two or more layers of felt throughout;[404] forming a shingle with the lower half laminated;[405] encasing the entire shingle in a layer of felt with a thick mastic in the center;[406] encasing the entire shingle in a layer of metal with a thick tapered mastic layer in the center.[407]

(2) *Shingles Surfaced with Mineral Granules*: Granules of a uniform color may be applied to the entire upper surface; or colored granules may be applied to that portion only of the upper surface which is intended to be exposed to the weather;[408] or applying granules of different colors in bands to the entire upper and

lower surfaces of the sheet;[409] or applying granules of different colors to the upper and lower halves respectively, on opposite sides of the sheet;[410] or granules of different colors may be applied to the lower halves of opposite sides, making the butt end thicker and reversible;[411] or granules of different colors may be applied to the upper and lower halves, respectively, of one side only, making the shingle reversible.[412] Variegated effects may be obtained by applying granules of two or more colors in stripes or irregular areas as described under "Decorative Roll-roofings"; or by pre-assorting shingles of different colors in the package so as to produce a mixture of colors on the roof when the shingles are laid.[413] Further colored effects may be obtained by applying bituminous paints in various shades to the granular surfacing and mixing the shingles on the roof.[414] Lastly, the weather surface may be corrugated to relieve it of its smooth appearance and to give it more texture.[415]

(3) *Rear Surface Coating*: This may be made of a low melting-point bituminous composition intended to cause the shingles to adhere together on the roof when laid,[416] known to the trade as a "seal back"; or conversely a non-adhesive backing may purposely be applied to prevent the shingles from adhering together in the package when shipped into hot climates;[417] or aqueous dispersions of asphalt may be used;[418] or metal soaps.[419]

(4) *Shingles Surfaced with Mineral Granules and Coated with Pigmented Hydraulic Cement*: Shingles may be made by surfacing an asphalt-saturated and coated sheet of rag-felt with uncolored mineral granules, which in turn are coated with a pigmented portland-cement grout [420] and allowed to set and harden in bundles. A magnesia-cement ("Sorel") grout has also been suggested for this purpose.[421]

(5) *Shingles Surfaced with Slate-veneer or Tile or Asbestos-cement*: Efforts have been made to combine prepared-roofing shingles with slates or roofing tile, by surfacing the former with a veneer of thinly split slate or a thin section of roofing tile,[422] or with a thin layer of asbestos-cement composition, but without much commercial success.

(6) *Shingles Surfaced with Metals, etc.*: Prepared-roofing shingles have been surfaced with metals, either by cementing a thin layer of sheet metal to the upper surface;[423] or else by electroplating the surface with copper,[424] or by spraying the surface with molten metals,[425] or with molten glass,[426] or with a pigmented synthetic resin composition.[427] Another alternative consists in affixing the layer of metal to the shingle with an interposed metallic layer of lower melting-point.[428] Shingles may also be edged with sheet-metal crimped into place.[429]

(7) *Shingles Reinforced with a Core of Sheet Metal or Wire Mesh*: It has been suggested that the shingles be stiffened by using a core of sheet metal or wire mesh, surfaced with bituminized fabric;[430] or by using a core of wire mesh filled with rubber and coated with asphalt surfaced in turn with mineral granules;[431] or by employing a core of sheet-metal coated with a bituminous composition and surfaced on one side with mineral granules;[432] but these expedients have not made much commercial headway.

(8) *Laminated Shingles*: Several layers of bituminized fabric may be cemented together with asphalt; or stapled together with wire to prevent separation;[433] or surfaced with subdivisions cut from a similar sheet to serve as patterns;[434] or may be constructed in various miscellaneous ways.[435]

(9) *Combinations with Asbestos Felt*: Attempts have been made to increase the fire-resistance of prepared roofing shingles through the use of asbestos felts, but these have so far proven unsuccessful.[436]

(10) *Combinations Involving the Use of Wooden Shingles*: Several different methods have been proposed to combine prepared-roofing shingles with wooden shingles;[437] or the use of wooden shingles impregnated or coated with bituminous compositions and surfaced with mineral granules;[438] or a similar structure fastened to a paper backing;[439] or asphalt shingles reinforced on the underside with wooden laths;[440] but such products have made little to no progress.

(11) *Miscellaneous Forms*: Other compositions involve the use of insulating board coated (but not saturated) with bituminous composition, which in turn is surfaced on one side with mineral granules or sheet metal.[441]

(A) Individual Shingles

These consist of shingle units cut in a single pattern, in distinction to the so-called strip-shingles which are cut in units having a repetition of the pattern so as to stimulate two or more individual shingles joined together. Individual shingles may be classified as follows:

(1) INDIVIDUAL SHINGLES FORMING RECTANGULAR PATTERNS WHEN LAID:

(*a*) *Butts free; shingles spaced closely together.*[442] These are customarily made in rectangular units measuring either 8 or 9 in. by 12¾ in. averaging 82 lb. per 108 sq. ft. known as standard weight, or else 10 or 12 in. by 16 in. averaging 105 lbs. per 108

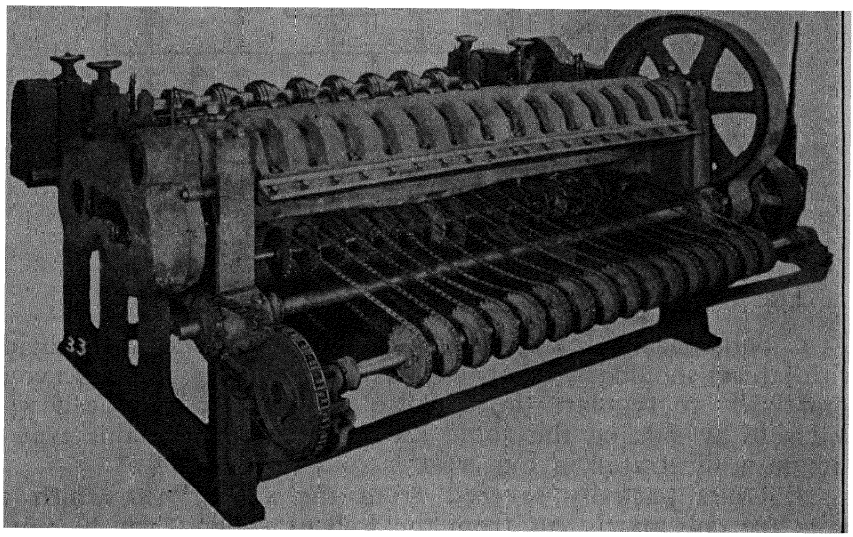
sq. ft. known as heavy weight, complying with the specifications given in Table LXX. The following quantities and weights are furnished per square, representing sufficient material to cover 100 sq. ft. of roof surface.

TABLE LXX

TYPES OF INDIVIDUAL SHINGLES

	Portion to be Exposed to the Weather			
	4-Inch		5-Inch	
Overall width, inches	8	9	10	12
Overall length, inches	12¾	12¾	16	16
To be spaced when laid, inches	½	½	¾	¾
Headlap, inches	4¾	4¾	5	5
Number shingles furnished per square	424	380	268	226
Square feet per square — Theoretical	300.0	302.0	297.7	301.2
Square feet per square — Actual	300.3	302.8	297.8	301.3
Weight per square, gross, pounds	235 ± 3 per cent		315 ± 4 per cent	

A machine for severing roll-roofing as it is manufactured into individual shingles is illustrated in Fig. 158.[448] The web is first cut into longitudinal strips by a gang of circular cutters mounted



Courtesy of Guyton & Cumfer Mfg. Co.

FIG. 158.—Cutter for Manufacturing Individual Shingles.

on a common shaft, and these in turn are severed logitudinally by a rapidly revolving fly-knife extending the full width of the cutter. The cutting blades may either actuate against the mineral surfacing, or against the reverse side of the sheet.[444] This type of shingle is illustrated in Fig. 159.

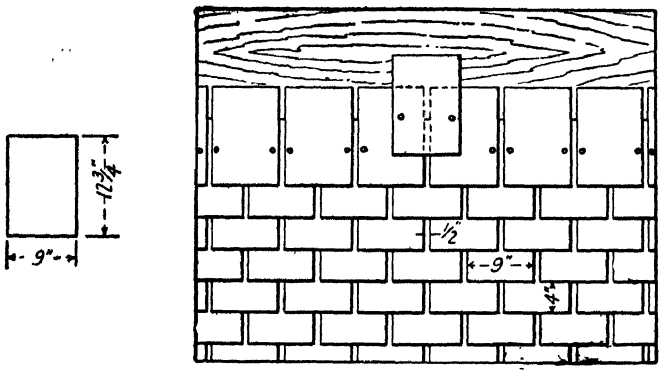


FIG. 159.—(Unpatented).

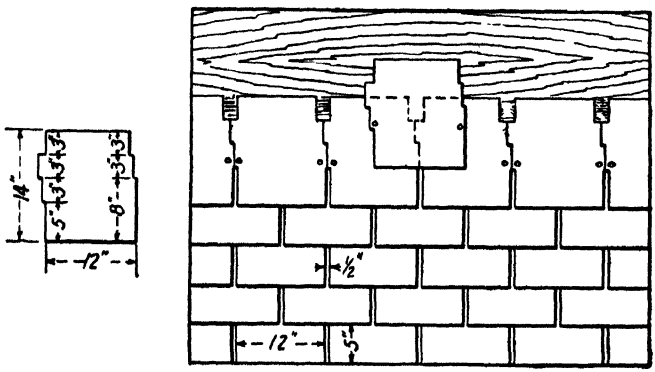


FIG. 160.—(U. S. Patent 1425922).

The following modifications have been proposed:

1. Self-spacing features,[445] in which the units are provided with lateral shoulders, or projections with recesses, which serve automatically to regulate the distance they are to be spaced when laid side by side on the roof. A representative of this type of shingle is illustrated in Fig. 160.

2. Units partially severed from the sheet,[446] in which the units are only partly cut from the web of roofing, which may be wound in roll form, and then separated by the applicator at the time they are to be applied to the roof; or else folded together in the package.[447]

3. Units L-shaped or inverted T-shaped.[448]

4. Units ⌂-shaped.[449]

(*b*) *Butts free; wide-spacing.* In this method of laying,[450] the units are spaced apart, as illustrated in Fig. 161. This procedure has the advantage of requiring less shingle area to cover 100 sq. ft.

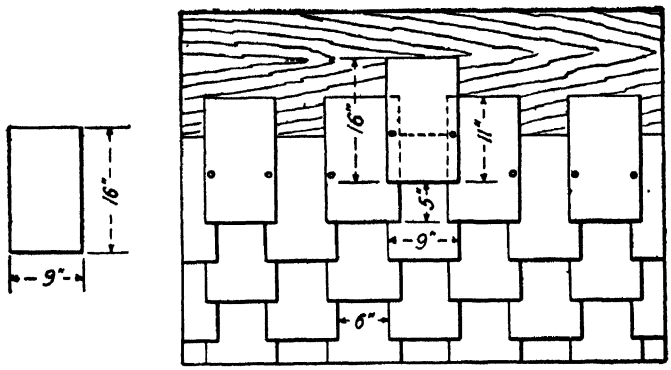


FIG. 161.—(U. S. Patent 1145440).

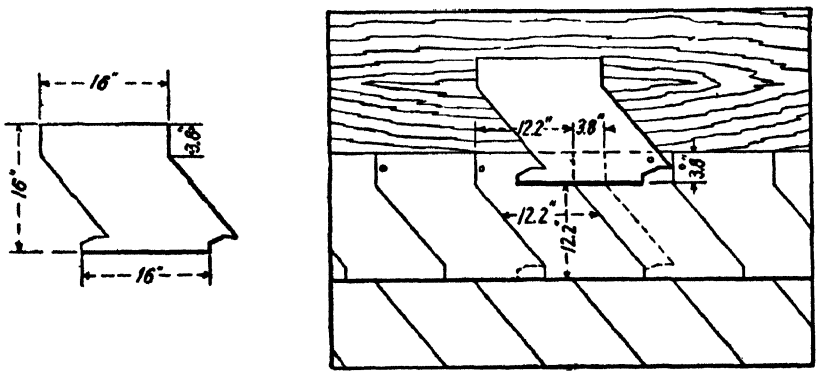


FIG. 162.—(U. S. Patent 1657082).

of roof surface. Thus, in the case of the shingle illustrated, only 192 sq. ft. of material are required per "square."

(*c*) *Butts secured; no metal fasteners; unfolded.* Numerous ingenious devices have been suggested for locking the shingles together when laid, so as to prevent the butts from turning upward,[451] either by the wind or otherwise; including the following:

1. Butts aligned in successive courses,[452] which is popularly known as the "Dutch-lap" method, one form of which (having sloping sides) is illustrated in Fig. 162.

2. Butts staggered in successive courses,[453] as illustrated in Fig. 163.

3. Forming ⌐¬-shaped patterns when laid,[454] as illustrated in Fig. 164.

(*d*) *Butts secured with metal fasteners; unfolded.* This involves the following alternates:

1. Butts aligned in successive courses.[455]
2. Butts staggered in successive courses.

(*e*) *Butts secured; no metal fasteners; folded.* This similarly involves the following alternates:

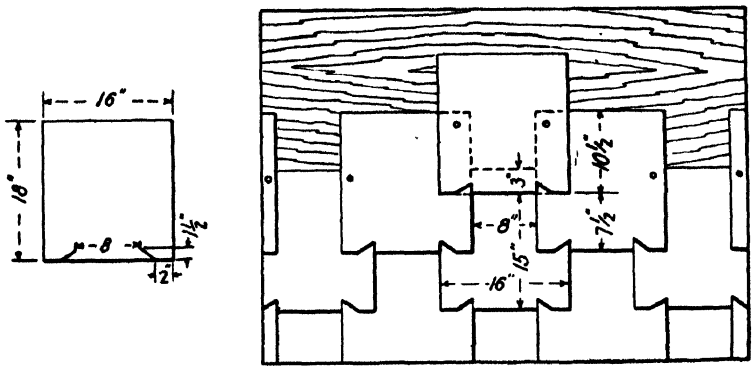


FIG. 163.—(U. S. Patent 1488447).

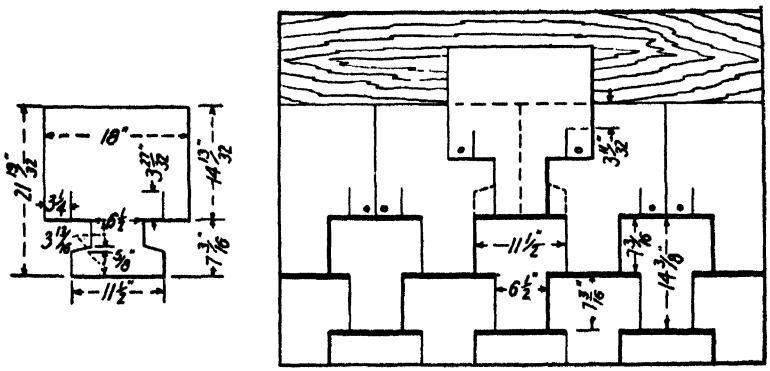


FIG. 164.—(U. S. Patent 1441420).

1. Butts aligned in successive courses.[456]
2. Butts staggered in successive courses.[457]

(*f*) *Butts secured with metal fasteners; folded.* Involving the following alternates:

1. Butts aligned in successive courses,[458] in which the butts are folded back beneath the shingle and fastened with nails or other metallic fasteners.

2. Butts staggered in successive courses.[459]

(*g*) *Marked forms.* In these, the shingles are provided with suitable guide marks to determine the proper exposure to the weather, as well as the correct distance that the shingles are to be spaced apart; or to accentuate the shadow lines when laid. The following alternatives have been proposed:

1. Darkened areas for guide marks,[460] consisting of paint, hot asphalt, or the like.

2. Darkened areas to serve for guide marks as well as shadow lines, as in the preceding.[461]

3. Raised ribs to serve for guide marks as well as for shadow lines.[462]

(*h*) *Embossed forms.* By embossing or raising portions of the shingle,[463] it may be made to appear more massive and to imitate vitrified tiles.

(*i*) *Laid in imitation of tiles.* Other expedients to add massiveness to the shingles consist in the following:

1. Laying over wooden battens.[464]

2. Laying over metal molding.[465]

3. Stiffening the shingle so that it will retain its contour unaided.[466]

(*j*) *Random-width forms.* In this case the units are furnished in varying widths and recommended to be laid with the butts out of alignment, so as to break up the monotony of the roof.

(2) INDIVIDUAL SHINGLES FORMING DIAMOND-SHAPED PATTERNS WHEN LAID:

Shingles of this type fall into the following general classes:

(*a*) *Butts free.*[467]

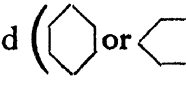
(*b*) *Butts secured; no metal fasteners; unfolded.*[468]

(*c*) *Butts secured with metal fasteners; unfolded.*[469] An example of this type is illustrated in Fig. 165.

(*d*) *Butts secured; no metal fasteners; folded.*[470]

(*e*) *Butts secured with metal fasteners; folded.*[471]

(3) INDIVIDUAL SHINGLES FORMING HEXAGONAL PATTERNS WHEN LAID:

Such hexagonal patterns may either have a point or a straight edge laid downward (), depending upon the particular construction of the shingle, and are classified as follows:

(*a*) *Butts free.*[472]

(*b*) *Butts secured; no metal fasteners; unfolded.*[473] Examples of this type are illustrated in Figs. 166, 167 and 168.

(*c*) *Butts secured with metal fasteners; unfolded.*[474]

(*d*) *Butts secured; no metal fasteners; folded.*[475]

(*e*) *Butts secured with metal fasteners; folded.*[476] A popular pattern of this class is illustrated in Fig. 169. This particular shingle is made in two sizes, measuring 16 by 16 in. and 12 by 12 in. overall respectively, consuming 146 and 172 sq. ft. material per

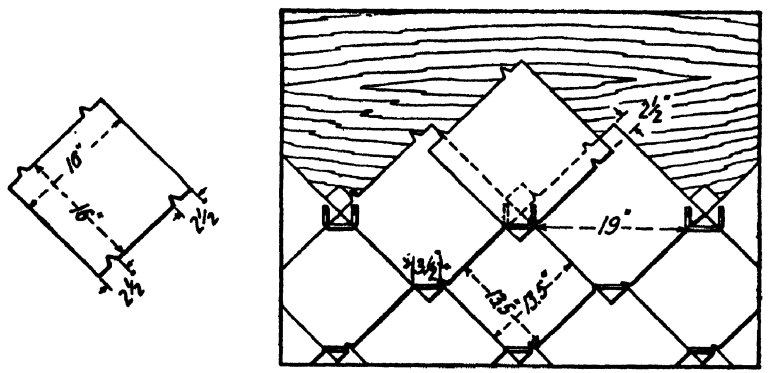


FIG. 165.—(U. S. Patent 1492516).

square. Both are laid with a 2½-in. headlap, and are recommended primarily for re-roofing work.

(*f*) *Butts secured with plastic bituminous cement.*[477]

(4) INDIVIDUAL SHINGLES FORMING OCTAGONAL PATTERNS WHEN LAID:

(*a*) *Butts free.*[478]
(*b*) *Butts secured; no metal fasteners; unfolded.*[479]
(*c*) *Butts secured with metal fasteners; unfolded.*[480]
(*d*) *Butts secured; no metal fasteners; folded.*[481]
(*e*) *Butts secured with metal fasteners; folded.*[482]

(5) INDIVIDUAL SHINGLES FORMING CIRCULAR OR CURVED EFFECTS WHEN LAID:

(*a*) *Butts free.*[483]
(*b*) *Butts secured; no metal fasteners; unfolded.*[484]
(*c*) *Butts secured with metal fasteners; unfolded.*[485]
(*d*) *Butts secured; no metal fasteners; folded.*[486]
(*e*) *Butts secured with metal fasteners; folded.*[487]

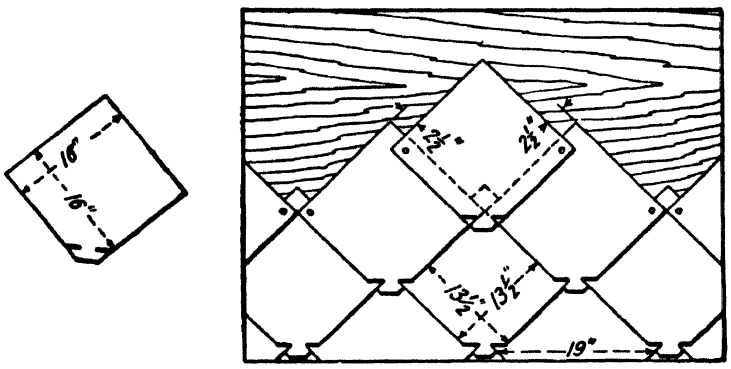


FIG. 166.—(U. S. Patent 1431103).

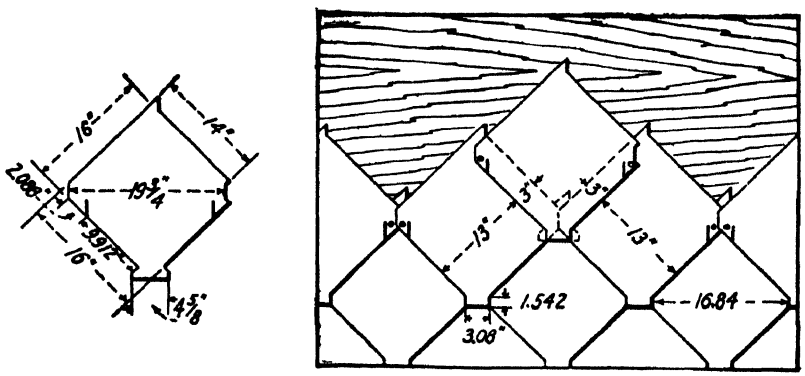


FIG. 167.—(U. S. Patent 1663658).

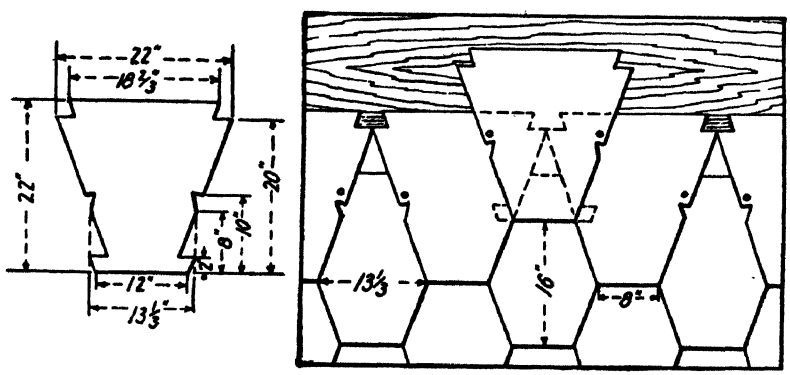


FIG. 168.—(U. S. Patent 1469041).

(6) INDIVIDUAL SHINGLES FORMING THATCHED EFFECTS WHEN LAID:

These are made with free butts,[488] and an example is given in Fig. 170. They may be finished in the following ways:

1. With brush-like filaments attached to the surface.[489]
2. With wires, cords, or asbestos strings attached to the surface.[490]

(7) REVERSIBLE FORMS OF INDIVIDUAL SHINGLES:

Shingles of this type fall into the following classes:

(*a*) *Forming either rectangular or diamond patterns.*[491]
(*b*) *Forming either rectangular or hexagonal patterns.*[492]
(*c*) *Forming either rectangular or octagonal patterns.*[493]
(*d*) *Forming either rectangular or circular patterns.*[494]
(*e*) *Forming either diamond or hexagonal patterns.*[495]
(*f*) *Forming either hexagonal or circular patterns.*[496]

(B) Strip-Shingles

These consist of two, three, or four units joined together in the form of flat strips, and are often termed "multiple shingle-strips." They have the advantage over individual shingles in that they may be laid more rapidly, and in certain cases consume a smaller quantity of material to cover 100 sq. ft. of roof surface. The appearance of the roof when laid is substantially the same as the corresponding class of individual shingles, and the patterns fall into similar groupings.

(1) STRIP-SHINGLES FORMING RECTANGULAR PATTERNS WHEN LAID:

(*a*) *Butts free; with cut-outs; close spacing.*[497]

Strip-shingles are of the general form illustrated in Fig. 171, showing four units joined together, although it is also customary to make strips of this type in two units, known as "twin-shingles," or in three units, known as "triplex-strips." They may either be cut across the web of material from which they are manufactured, or in a direction measured longitudinally with respect to the web. Such strips are usually made in three sizes, complying with the specifications given in Table LXXI on page 670.

A cutter for producing this pattern of strip-shingles is shown in Fig. 172, which serves not only to sever the strips from the web, but to count and stack them as well.

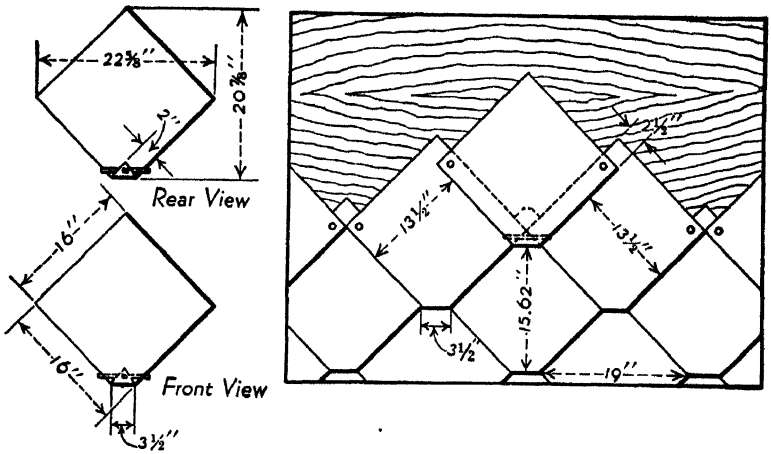


FIG. 169.—(U. S. Patent 1274410).

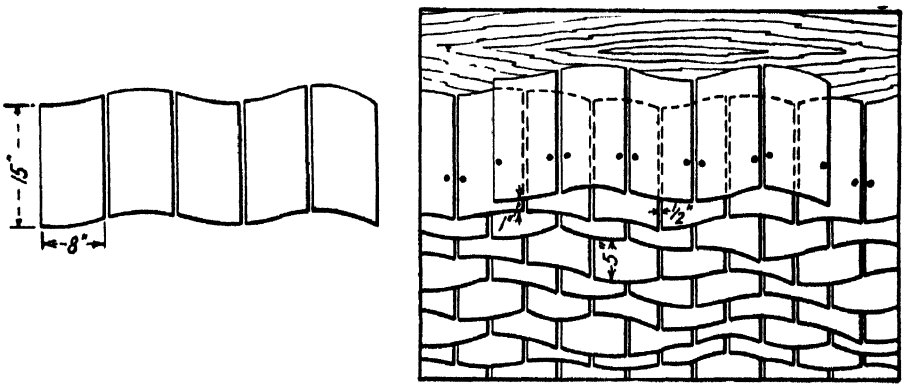


FIG. 170.—(U. S. Patent 1596880).

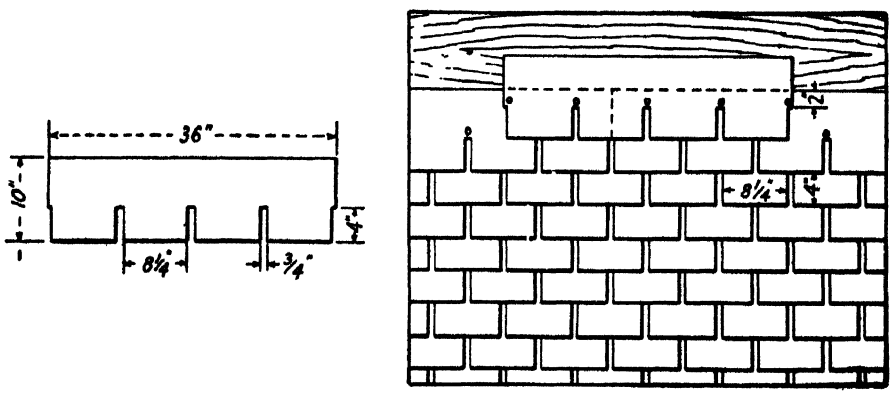


FIG. 171.—(U. S. Patent 1150298).

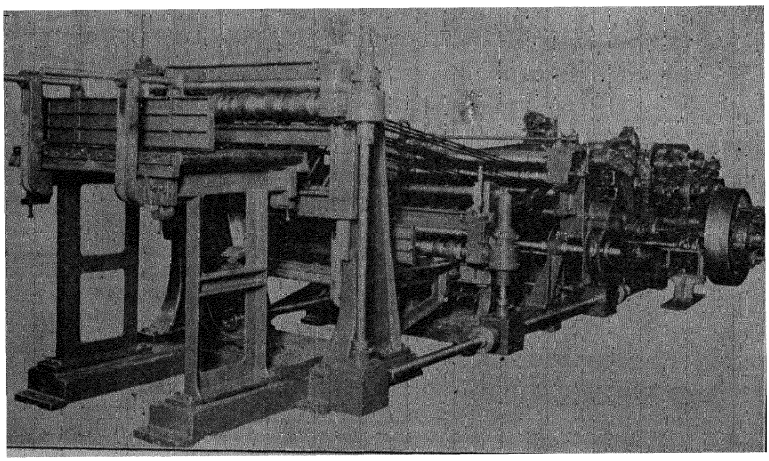
TABLE LXXI

TYPES OF SQUARE-BUTT STRIP-SHINGLES

	Portion to be Exposed to the Weather					
	4 Inches				5 Inches	
Overall width, inches	10	10	12½	12½	12	12
Overall length, inches	36	32	36	32	36	32
Width of cut-outs, inches	¾	¾	¾	¾	¾	¾
Headlap, inches	2	2	4½	4½	2	2
Number of shingles furnished per square	100	112	100	112	80	90
Square feet per square — Theoretical	250.0	250.0	312.5	312.5	240.0	240.0
Square feet per square — Actual	250.0	249.0	312.5	311.1	240.0	240.0
Weight per square, pounds gross	200±3 per cent		250±3 per cent		250±4 per cent	

This form of strip-shingle may be modified as follows:

1. With diagonal or curved cut-outs.[498]
2. With partially severed cut-outs.[499]
3. With transverse incisions capable of forming cut-outs when the shingle is drawn apart longitudinally.[500]



Courtesy of The Samuel Langston Co.

FIG. 172.—Cutter for Manufacturing Strip-shingles.

4. With the edges adjacent to cut-outs raised or folded.[501]
5. With the edges of the butts bent upward.[502]
6. With the edges torn irregularly.[503]
7. With the lateral ends cut diagonally.[504]

8. With self-spacing features.[505]
9. With the butts doubled underneath.[506]
10. Laminated forms.[507]
11. Partially severed from the sheet.[508]
12. Cut in staggered form.[509]

(*b*) *Butts free; without cut-outs.*[510] This type has not proven practical.

(*c*) *Butts free; wide spacing.*[511] A representative of this form of strip is illustrated in Fig. 173. Modifications of this type include the following:

1. With the lateral ends cut diagonally.[512]
2. With self-spacing features.[513]

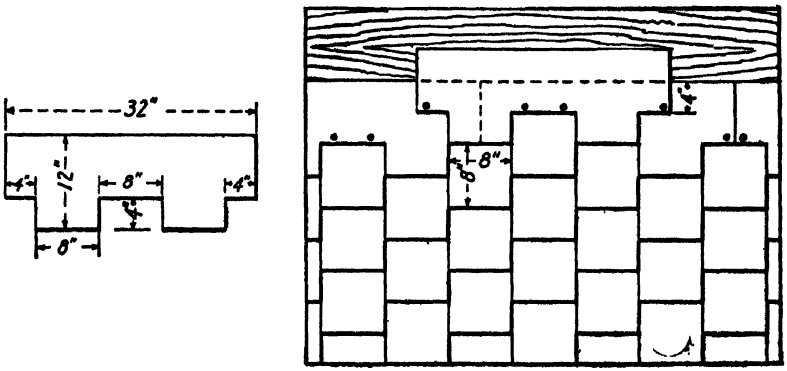


FIG. 173.—(U. S. Patent 1629146).

3. With the upper and lower edges replicas of each other.[514]
4. Secured with metal fasteners.[515]

(*d*) *Butts secured; no metal fasteners; unfolded.* This involves the following alternates:

1. Butts aligned in successive courses.[516]
2. Butts staggered in successive courses,[517] as illustrated in Fig. 174.
3. Forming ⌐⌐-shaped patterns when laid.[518]

(*e*) *Butts secured with metal fasteners; unfolded.* This involves the following alternates:

1. Butts aligned in successive courses.[519]
2. Butts staggered in successive courses.[520]

(*f*) *Butts secured; no metal fasteners; folded.* This involves the following alternates:

1. Butts aligned in successive courses.[521]
2. Butts staggered in successive courses.[522]

(*g*) *Butts secured with metal fasteners; folded.* This involves the following alternates:

1. Butts aligned in successive courses.[523]
2. Butts staggered in successive courses.[524]

(*h*) *Marked forms.* This type involves the use of markings to simulate cut-outs, or to accentuate same, and includes the following variations:

1. Surfacing depressed in stripes or areas,[525] as illustrated in Fig. 175.

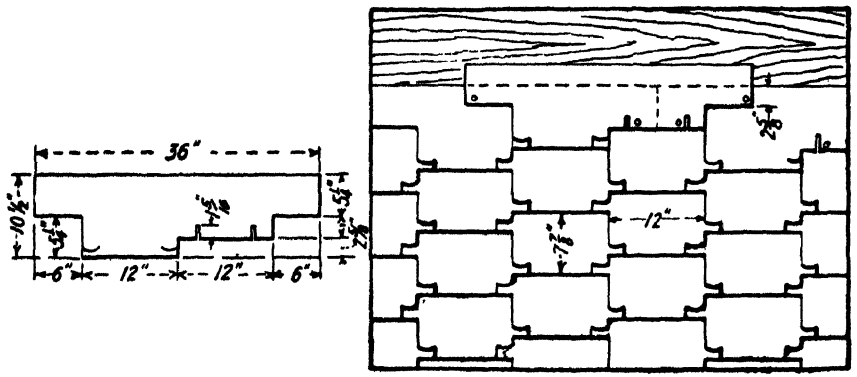


FIG. 174.—(U. S. Patent 1607357).

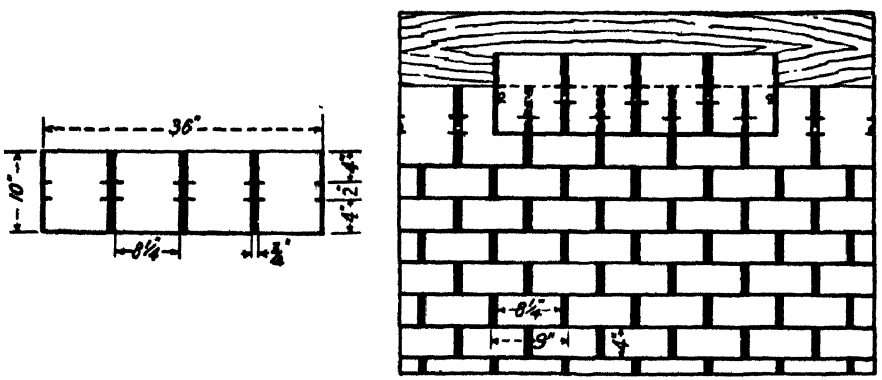


FIG. 175.—(U. S. Patent 1296984).

2. Surfacing omitted or removed in stripes or areas.[526]
3. Surfacing of contrasting color in stripes or areas.[527]
4. Painted stripes or areas.[528]
5. Superimposed stripes or areas.[529]
6. Perforations to simulate cut-outs.[530]

(*i*) *Embossed forms.*[531] This type involves the following procedures:

1. Fluting the lower edge of the shingle.[532]
2. Filling in with wooden battens underneath the shingle.[533]

(*j*) *Folded together in package.*[534]
(*k*) *Random-width forms.*[535] Illustrated in Fig. 176.
(*l*) *Laid in imitation of tiling over wooden battens.*[536]
(*m*) *Creased or folded forms.*[537]

(2) STRIP-SHINGLES FORMING DIAMOND-SHAPED PATTERNS WHEN LAID:

(*a*) *Butts free:*
1. Without cut-outs;[538]
   α. Straight upper edge.
   β. Diamond-shaped upper edge, as illustrated in Fig. 177.

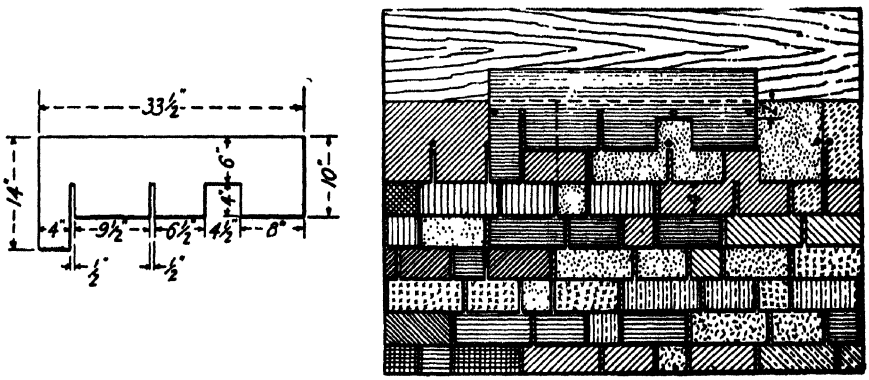


FIG. 176.—(U. S. Patent 1495070).

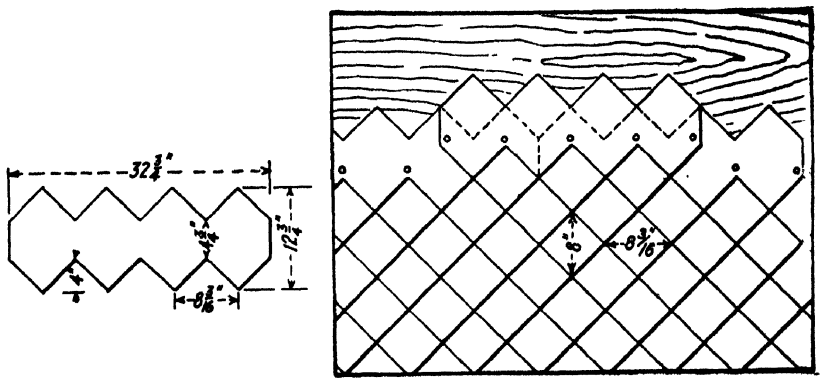


FIG. 177.—(U. S. Patent 1243064).

2. With cut-outs,[539] as illustrated in Fig. 179.
3. Marked forms.[540]
4. Cut in staggered form.[541]

(*b*) *Butts secured; no metal fasteners; unfolded.*[542]
(*c*) *Butts secured with metal fasteners; unfolded.*[543]

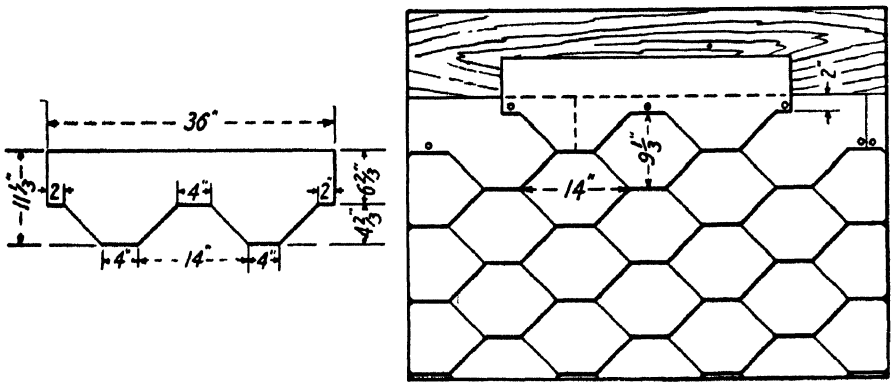


FIG. 178.—(U. S. Patent 1604339).

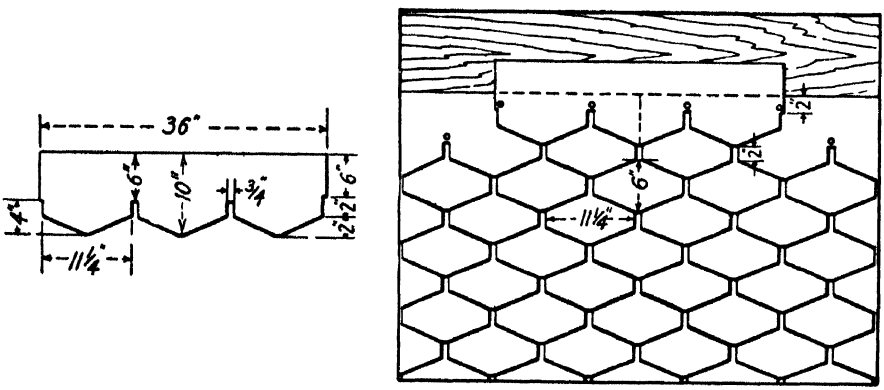


FIG. 179.—(U. S. Patent 1732403).

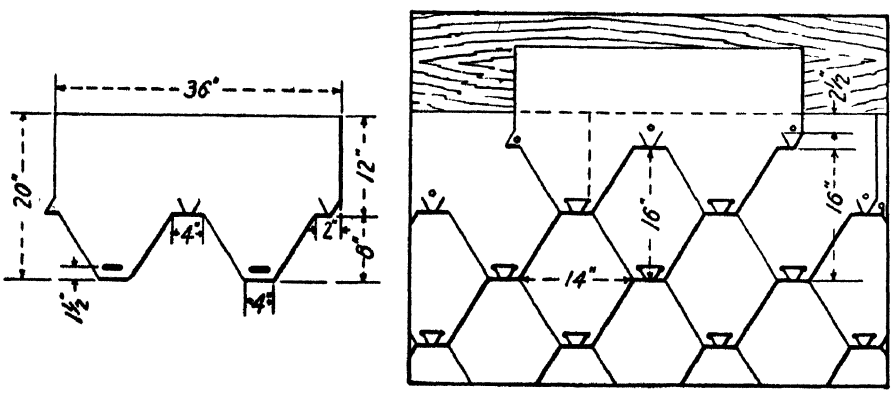


FIG. 180.—(U. S. Patent 1729846).

(3) STRIP-SHINGLES FORMING HEXAGONAL PATTERNS WHEN LAID:

(*a*) *Butts free; without cut-outs.*[544] This form has met with considerable popularity, and may be made with equilateral sided tabs, or else the lower edge may be narrower than the adjacent sloping sides, as illustrated in Fig. 178.

These strips are made in a multitude of dimensions, as shown in Table LXXII on page 676.

This type also includes the following variations:

1. Ends cut diagonally.[545]
2. One side of tab cut vertically, or irregularly.[546]
3. Shingle cut in staggered form.[547]

(*b*) *Butts free; with cut-outs.*[548] In this type the lower edge is pointed, as illustrated in Fig. 179.

(*c*) *Butts secured; no metal fasteners; unfolded.*[549] Typical examples of this type are shown in Figs. 180 and 181.

(*d*) *Butts secured with metal fasteners; unfolded.*[550]

(*e*) *Butts secured; no metal fasteners; folded.*[551] A specimen representing this type is shown in Fig. 182.

(*f*) *Butts secured with metal fasteners; folded.*[552]

(*g*) *Marked forms.*[553]

(*h*) *Creased or folded forms.*[554]

(*i*) *Plurality of strips in staggered relation.*[555]

(4) STRIP-SHINGLES FORMING OCTAGONAL PATTERNS WHEN LAID:

(*a*) *Butts free; with cut-outs; close spacing.* This form is attractive in appearance and may be manufactured more economically than the rectangular type, although not as economically as the hexagonal type, consuming in the neighborhood of 230 sq. ft. of material per square. This type includes the following variations:

1. Having a straight upper edge.[556]
2. Having nailing-tabs along the upper edge located centrally over the butts,[557] as illustrated in Fig. 183.
3. Having nailing-tabs along the upper edge located over the cut-outs.[558]
4. Having the lateral ends cut diagonally.[559]
5. The strip being cut in staggered form.[560]

(*b*) *Butts free; without cut-outs; wide spacing.*[561] A strip of this group is shown in Fig. 184.

(*c*) *Butts secured; no metal fasteners; unfolded.*[562]

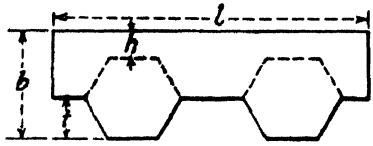
(*d*) *Butts secured with metal fasteners; unfolded.*[563]

(*e*) *Butts secured; no metal fasteners; folded.*[564]

(*f*) *Butts secured with metal fasteners; folded.*[565]

TABLE LXXII

Types of Hexagonal Strip-Shingles



t = length of tabs in inches;
b = overall width of strip in inches;
l = length of strip in inches;
h = headlap in inches $= b - 2t$;

E = exposed area of strip in sq. ft. $= \frac{tl}{144}$;

N = number of strips to be furnished per "square" $= \frac{100}{E}$;

T = total area per strip in sq. ft. $= \left(b - \frac{t}{2}\right)\left(\frac{l}{144}\right)$;

A_1 = theoretical area per "square" in sq. ft. $= \left(b - \frac{t}{2}\right)\left(\frac{100}{t}\right)$;

A_2 = actual area per "square" in sq. ft. $= \left(b - \frac{t}{2}\right)\left(\frac{lN}{144}\right)$.

Length of Tab (Ins.)	Overall Width (Ins.)	Overall Length (Ins.)	Headlap (Ins.)	Exposed Area per Strip (Sq. Ft.)	Strips per Square (Number)	Total Area per Strip (Sq. Ft.)	Area per "Square"	
							Theoretical (Sq. Ft.)	Actual (Sq. Ft.)
t	b	l	h	E	N	T	A_1	A_2
4	10	32	2	0.889	112	1.778	200	199
	12	32	4	0.889	112	2.222	250	249
	10	36	2	1.000	100	2.000	200	200
	12	36	4	1.000	100	2.500	250	250
	$12\frac{1}{2}$	36	$4\frac{1}{2}$	1.000	100	2.625	$262\frac{1}{2}$	$262\frac{1}{2}$
	$12\frac{2}{3}$	36	$4\frac{2}{3}$	1.000	100	2.6675	$266\frac{3}{4}$	$266\frac{3}{4}$
$4\frac{1}{2}$	$11\frac{1}{4}$	36	$2\frac{1}{4}$	1.125	90	2.25	200	$202\frac{1}{2}$
	$13\frac{1}{4}$	36	$4\frac{1}{4}$	1.125	90	2.75	$244\frac{4}{9}$	$247\frac{1}{2}$
$4\frac{2}{3}$	$11\frac{1}{3}$	36	2	1.167	86	2.25	$192\frac{6}{7}$	$193\frac{1}{2}$
	$12\frac{1}{3}$	36	3	1.167	86	2.50	$214\frac{2}{7}$	215
5	12	36	2	1.250	80	2.375	190	190
	$12\frac{1}{2}$	36	$2\frac{1}{2}$	1.250	80	2.500	200	200
$5\frac{1}{4}$	$13\frac{1}{8}$	36	$2\frac{5}{8}$	1.3125	76	2.625	200	$199\frac{1}{2}$
	$13\frac{1}{4}$	36	$2\frac{3}{4}$	1.3125	76	2.656	202.4	202
	$13\frac{1}{2}$	36	3	1.3125	76	2.719	207.1	$206\frac{1}{2}$

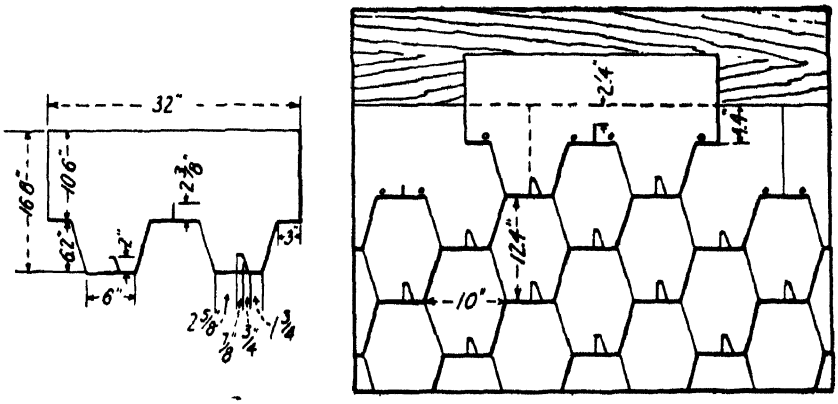


FIG. 181.—(U. S. Patent 1524125).

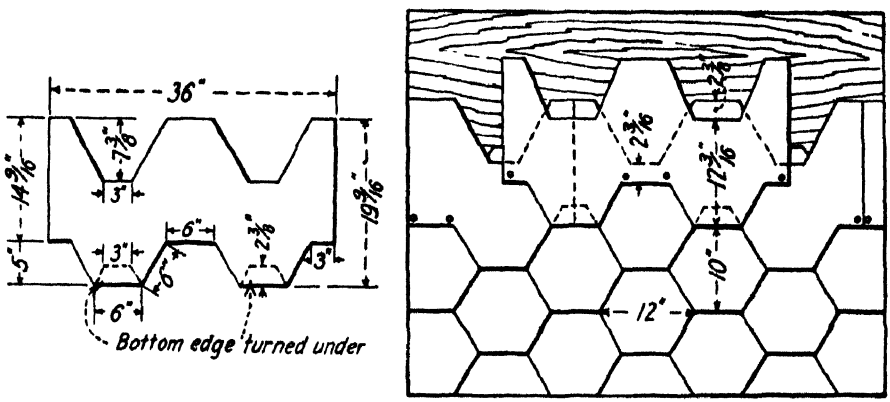


FIG. 182.—(U. S. Patent 1402361).

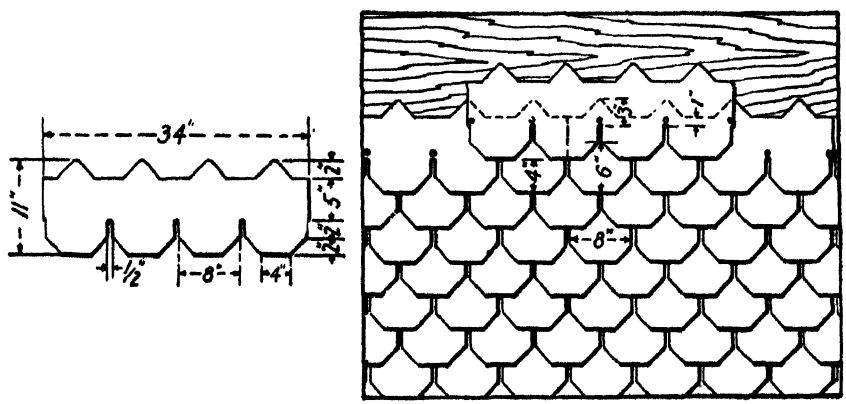


FIG. 183.—(U. S. Patent 1326899).

(*g*) *Marked forms.*[566]
(*h*) *Creased or folded forms.*[567]

(5) STRIP-SHINGLES FORMING CIRCULAR OR CURVED EFFECTS WHEN LAID:

(*a*) *Butts free; with cut-outs.*[568]
(*b*) *Butts free; without cut-outs.*[569] This type is illustrated in Fig. 185.
(*c*) *Butts secured; no metal fasteners; unfolded.*[570]
(*d*) *Butts secured with metal fasteners; unfolded.*[571]
(*e*) *Marked forms.*[572]

(6) STRIP-SHINGLES FORMING THATCHED EFFECTS WHEN LAID:

(*a*) *Butts free; with cut-outs.*[573] This type is illustrated in Fig. 186.
(*b*) *Butts free; without cut-outs.*[574]
(*c*) *Butts secured; without cut-outs.*[575]
(*d*) *Marked forms.*[576]

(7) STRIP-SHINGLES FORMING MISCELLANEOUS PATTERNS WHEN LAID:

(*a*) *Combinations of rectangular and octagonal patterns.*[577]
(*b*) *Combinations of diamond and octagonal patterns.*[578]
(*c*) *Combinations of hexagonal and diamond patterns.*[579]
(*d*) *Combinations of hexagonal and circular patterns.*[580]
(*e*) *Combinations of diamond, hexagonal and circular patterns.*[581]
(*f*) *Variations of octagonal pattern.*[582]
(*g*) *Variations of diamond pattern.*[583]
(*h*) *Variations of hexagonal pattern.*[584]

(8) REVERSIBLE FORMS OF STRIP-SHINGLES:

These have the two long edges cut in different patterns respectively, so that a roof of one design will be obtained if laid with one edge downward, and another pattern if the strip is reversed top for bottom. They fall into the following groups:

(*a*) *Forming either rectangular or diamond patterns.*[585]
(*b*) *Forming either rectangular or hexagonal patterns.*[586]
(*c*) *Forming either rectangular or octagonal patterns.*[587]
(*d*) *Forming either rectangular or circular patterns.*[588]
(*e*) *Forming either diamond or hexagonal patterns.*[589]
(*f*) *Forming either diamond or octagonal patterns.*[590]

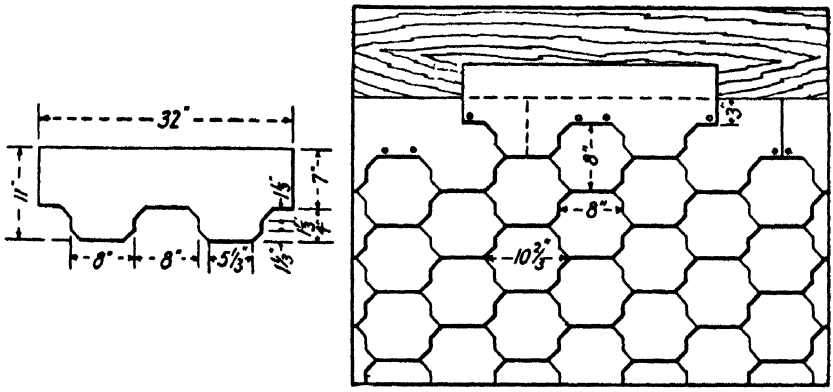


FIG. 184.—(U. S. Patent 1533969).

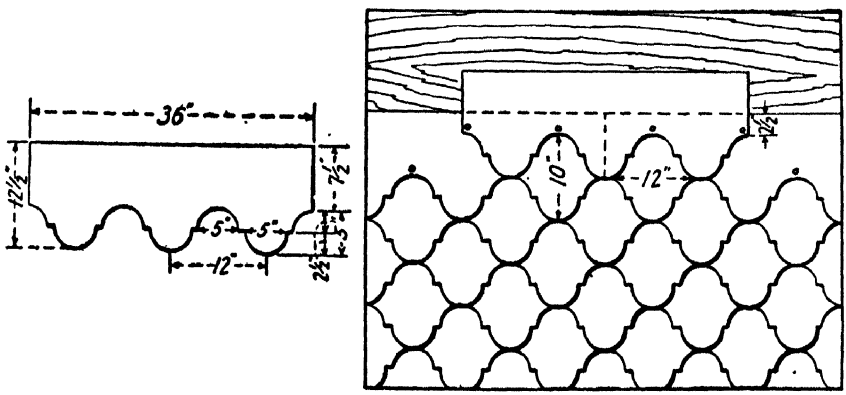


FIG. 185.—(U. S. Design Patent 67565).

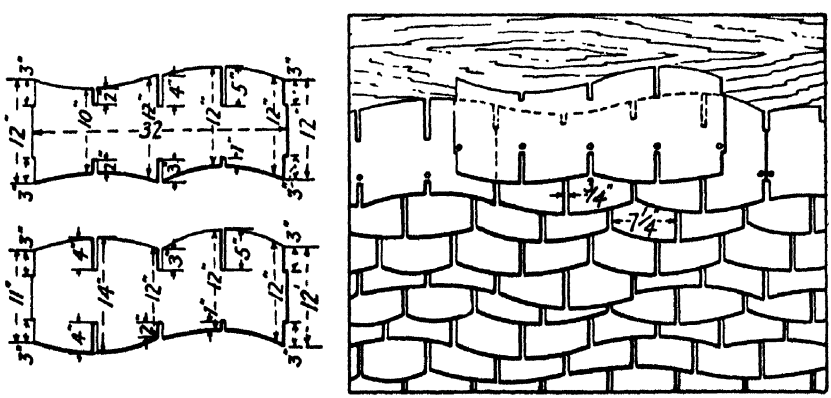


FIG. 186.—(U. S. Patent 1688917).

The figures in Table LXXIII show the number of shingles or shingle-strips furnished per square, the area of material supplied per square, the gross weight per square, and the headlap of the

TABLE LXXIII

DATA PERTAINING TO SHINGLES SHOWN IN FIGS. 159 TO 186 INCLUSIVE (EXCL. FIG. 172)

Figure Number	Number Shingles per Square	Area per Square (Sq. Feet)	Gross Weight per Square (Pounds)	Headlap (Inches)	Remarks
159	380	302	240	4¾	For other sizes see text.
160	228	266	215	4	
161	192	192	155	1	
162	98	174	140	3.8	
163	80	160	130	3	When exposed 7½ in.
	100	200	160	6	When exposed 6 in.
	133	266	200	9	When exposed 4½ in.
164	144	258	210	3¼	
165	82	146	115	2.5	
166	82	146*	115	2.5	Lower corner cut off.
167	86	153	125	3	
168	84	197	155	4	
169	82	146	115	2.5	For other sizes see text.
170	564	470	375	6–14	Exposure varies from 1 to 5 in. (averaging 3 in.)
171	100	250†	200	2	For other sizes see text.
173	112	250	200	4	
174	100	250	200	2	
175	160	315	250	2⅝	
176	100	250†	200	2	
177	112	222.8*	180	4¾	
178	86	193	155	2	For other sizes see text.
179	100	225*†	180	2	
180	50	200	160	2½	
181	72	219	175	2	
182	78	280	225	2 3/16	
183	108	223.2*†	180	2	
184	112	225	180	3	
185	80	200	160	3	
186	150	400	320	2–5	Exposure varies from 1 to 5 in. (averaging 3 in.)

* Not including waste in cutting.
† Not including loss of cut-outs.

particular shingles illustrated in Figs. 159 to 186 inclusive, predicated on the supposition that they are all cut from the same basic roofing material weighing an average of 82.0 lb. per 108 sq. ft. net.

(C) Asphalt Sidings

Sidings of this character are intended to take the place of ordinary wooden clap-boards on the sides of wooden houses and are claimed to be more durable and require less up-keep. In some cases asphalt shingles are recommended for this purpose, particularly those forms which lock together at the butts. The most popular form consists of a modified type of strip shingle surfaced with mineral granules applied to the surface, the lower portion carrying a second coating of asphalt in which granules of a different color are embedded, so as to simulate the appearance of tapestry bricks when laid.[591] This particular form is illustrated in Fig. 187.

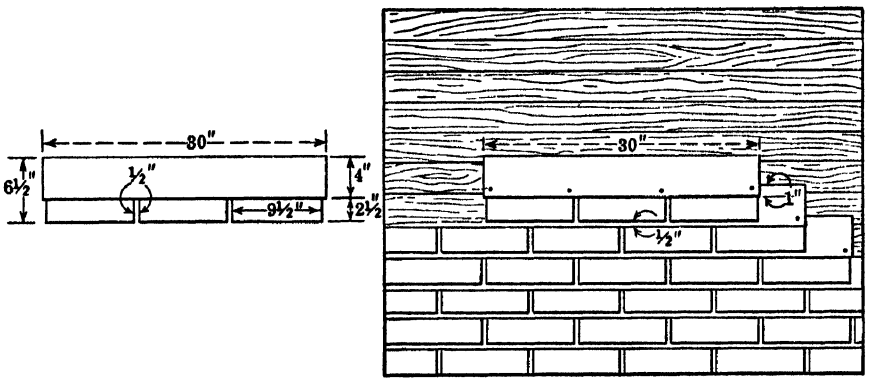


FIG. 187.—Asphalt Siding.

Other modifications include: using long narrow strips of granular surfaced roofing, which may be made tapered;[592] long narrow strips of granular surfaced roofing attached to the lower end, as an appendage to wooden boards;[593] or as an appendage to strips of Portland cement;[594] strips of granular-surfaced prepared roofing finished in imitation of bricks cemented to a rigid base of wall-board;[595] strips of granular-surfaced prepared roofing enclosing a tapered core of fibrated asphalt;[596] wire mesh pressed to imitate bricks and waterproofed with asphalt surfaced with mineral granules;[597] asbestos-cement tiles cemented with asphalt to a bituminized-felt base;[598] thin facing-bricks cemented with asphalt to a bituminized-felt base;[599] etc.

Fastening Devices. Sheets of prepared roofing were originally fastened to the roof boards by means of ⅞ to 1-in. steel nails, driven through convex discs composed of American terne plate (thickness IX), ⅞ to 1 in. in diameter, the nails being spaced 2 in. from centers.

Discs formed of prepared roofing,[600] square caps of enameled metal having a raised center portion, [601] steel discs fastened directly to the tops of wire nails were next proposed.[602]

At the present time the "caps" have been almost entirely replaced by large-headed roofing nails. For the better grades of roofing and shingles the nails should either be sherardized or galvanized, and protected with not less than 37½ mg. zinc per square centimeter, corresponding to 1¼ oz. per square foot of surface. Sherardizing is preferable to galvanizing, as it affords better protection to the steel, assuming that equal weights of zinc are applied in both cases, and forms a coating that will not chip or flake off should the nail shank or head bend. This is very likely to occur at the junction of the shank and head when the nail is driven in place. A well-sherardized nail will withstand the weather under normal conditions from ten to fifteen years, without further protection, and in the author's opinion constitutes the most satisfactory means of rust-proofing.[603] A coating of cadmium, applied by electrolysis has also been recommended.

With cheaper prepared roofings, it is customary to furnish the nails in the "bright" form, i.e., without being treated, but it then becomes necessary to protect the heads when hammered into place, with a liberal coating of "lap cement." Although the latter will tend to *retard* corrosion of the steel, the practice is not to be recommended, where the durability of the roof is an important consideration.

Various forms of nails have been suggested, including large-headed nails having grooves or sharp projections provided on the underside of the head;[604] nails having grooves on the shank;[605] nails with threaded shanks;[606] wires bent or shaped in sundry forms,[607] etc.

In fastening the seams of roll-roofings, it is customary to spread so-called "lap-cement" between the laps before the nails or other fastening devices are driven into place. A tape coated with an

adhesive bituminous composition has been suggested as a substitute for the lap-cement, to be inserted between the seams;[608] also the use of an adhesive surface-coating applied to the edge of the sheet where the joint is to be formed;[609] also moistening the seams with a petroleum distillate to render same adhesive.[610]

The following specifications have been proposed for lap-cement and nails to be furnished with roll-roofings:[611]

Lap-cement. Flash-point min. 80° F. by the Tag. closed-tester (Test 17*c*); quantity to be furnished per square for roofings 36 in. wide ¾ pint per square, 32 in. wide 1 pint, 18 and 16 in. wide 1¼–2 pints.[612]

Nails. Diameter shank: max. 0.135 in., min. 0.105 in., either smooth or barbed (in which case the barbings shall not increase the diameter more than 0.001 in.). Length of shank: min. ¾ in. to max. 1 in. Heads: min. diameter ⅜ in., min. thickness 0.025 in. Coating: shall be zinc, applied by hot galvanizing, electro-galvanizing, or sherardizing, in an amount of not less than ½ oz. nor more than 1¼ oz. per sq. ft. Quantity: the minimum count per square shall be 252 for roll-roofings 36 in. wide, 275 for 32-in. roll-roofings, 504 for 18-in., and 550 for 16-in. (these quantities are sufficient for one long seam and one end seam with the nails spaced 2 in.).

The addition of 5 to 20 per cent by weight of short-fibered asbestos to lap-cement may be permitted, in which event the quantity of cement should be increased by ¼ pint per "square." Special tools have been devised for conveniently applying the lap-cement.[613]

Shingles are ordinarily fastened with nails, the specifications of which correspond to those applicable to roll-roofings, except that they shall be 1 in. long when laid on solid sheathing, and 1½ to 1¾ in. long when laid over old shingles. The number of nails required will depend upon the size and form of the particular shingle. Alternate procedures consist in cementing down the shingles with a plastic bituminous cement;[614] or by applying patches of combustible material to the shingles, which when ignited (sic.) will soften the underlying asphalt coating and cause adhesion.[615]

Metal cleats for roll-roofings. A number of devices have been proposed for this purpose, including flat strips of metal fastened over the seams;[616] flat strips of metal fastened in between the

seams;[617] strips of metal with spaced perforated concave portions;[618] strips reinforced by longitudinal ribs;[619] wires bent or shaped in various forms;[620] etc.

Concealed nailing for roll-roofings. Various methods have been devised for forming the seams of roll-roofings and at the same time protecting the nail heads, by covering the nail heads with flaps, folding over the roofing,[621] etc.

Forming standing seams. Another expedient consists in laying the roll-roofing over wooden battens to break up the surface into a number of standing seams or elevated portions intended to cast shadows, or to give the appearance of tiles;[622] or laying the shingles between raised battens which also serve as supporting elements.[623]

Methods of Packaging. Roll-roofings are prepared for shipment by winding them on the outside with a heavy paper "wrapper," and packing the "fixtures" (consisting of the nails and can of lap-cement) in the core of the roll. The insertion of a rip-cord in the wrapper to facilitate tearing it off has also been suggested.[624]

Various devices have been used for holding the fixtures in place, including sheets of cloth or heavy paper pasted over the ends; introducing wire springs into the ends of the core;[625] cylindrical plugs of wood driven in the openings at the end of the roll and fastened together with wire;[626] mushroom-shaped plugs inserted in the ends of the roll;[627] using a paper wrapper wider than the roll, turning over the edges into the core and wedging into place with cylindrical plugs;[628] sealing the openings of the core with plaster-of-Paris plugs;[629] constricting the openings of the core by driving wooden wedges between the convolutions at the ends of the roll;[630] covering the ends of the roll with wooden discs wired together;[631] covering the ends with metal discs wired together;[632] covering the ends with metal discs fastened together with a light metal rod;[633] metal discs held in place by projecting flanges;[634] metal discs held in place by means of curved springs;[635] packing the fixtures in an elongated cylindrical cardboard case, which at the same time serves as a mandrel for the roll;[636] fastening the fixtures in the core with a wire clip;[637] wrapping the nails and can of lap-cement in a paper or cloth package provided with a flap which is inserted between the inner convolutions of the roll of roofing as the sheet is wound on the machine;[638] using the innermost convolution of the roll for the

same purpose;[639] enclosing the entire roll in a closely fitting cylindrical receptable composed of fiber-board;[640] packing the fixtures in a device held in place with projecting springs;[641] etc. At the present time the usual practice consists in fastening the ends of the roll with muslin pasted in place, or by using metal end protectors fastened together with wires or rods.

Shingles are packed in bundles protected on the top and bottom with wood veneer, plain[642] or asphalt fiber-board,[643] which are fastened in place either by means of wires[644] or metal tapes. An alternate procedure consists in packing the shingles in cardboard cartons.[645]

Methods of Laying Roofings and Shingles. Roofings may either be laid on the roof in a single course, or in a so-called "built-up" form, composed of two or more bituminated sheets constructed on the roof of the building. The single-course method is adapted only for application over wood, whereas built-up roofs are equally suitable for use over wooden, gypsum or concrete roof decks.

Laying the Fabrics in a Single Course. In this case the sheets are laid with 2-in. seams and cross-seams, the joints being sealed with liquid lap-cement, and fastened with large headed nails, or with any of the other devices described. A roller-guide for correctly spacing the nails has been patented.[646] To economize in the use of material it has been suggested that the seams may be made wider at the base of the roof than at the eaves.[647]

Where the sheet is surfaced with moderately fine mineral matter on but one side, it is advisable to lay this *against* the roof boards.[648] A single course of multiple-layered roofing, composed of burlap cemented between two layers of asphalt-saturated and coated felt is introduced between two layers of sheathing boards for covering freight cars, the function of the upper layer being to protect the roofing from injury.[649] A single layer of prepared roofing may also be cemented fast to metal railway car roofs.[650] It has been suggested that the roof boards be painted with sodium silicate,[651] or that a combination of roofing with an oil-proof sheet[652] be used to prevent the resinous bodies in the wood from adversely affecting the bituminous constituents of the roofing.

Roll-roofings may be laid over old wooden shingle roofs, by first affixing nailing strips to fall underneath the seams.[653]

Built-up Roofs.[654] These may be laid over wood, concrete or gypsum roof decks in 3, 4, or 5 layers or plies as indicated in Table LXXIV. Either tarred-felt and coal-tar pitch, or asphalt-saturated felt and asphalt may be used. The first produces what is known as a "tar roof" and the second an "asphalt built-up roof." Either type may be surfaced with slag or gravel, or where a particularly durable form of construction is desired, the finish may consist of tiles embedded in a bed of asphalt mastic (used in the early days), or embedded in Portland-cement mortar [655] in lieu of

TABLE LXXIV

BUILT-UP ROOFS—WEIGHTS OF COMPONENTS PER 100 SQ. FT. ROOF AREA

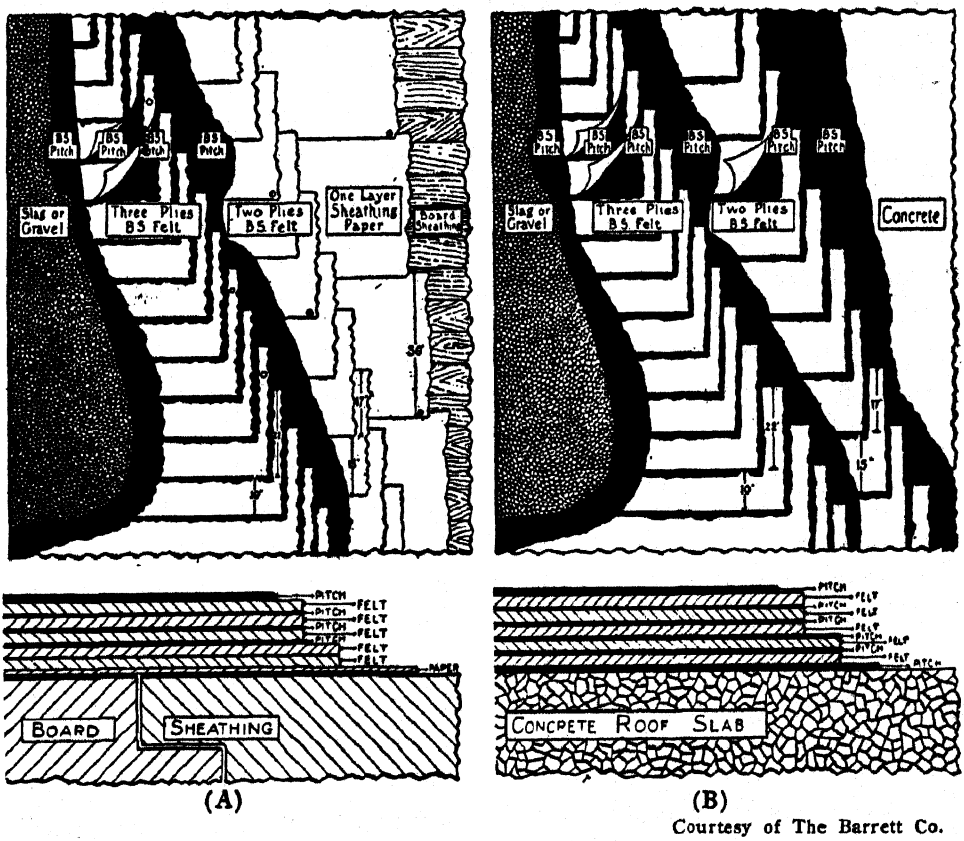
	5-Ply				4-Ply				3-Ply	
	Over Wood		Over Concrete		Over Wood		Over Concrete		Over Concrete	
	Tar[656]	Asphalt[657]	Tar[658]	Asphalt[659]	Tar[660]	Asphalt[661]	Tar[662]	Asphalt[663]	Tar[664]	Asphalt[665]
Paint primer (asphaltic)				7-5*				7-5*		7-5*
Sheathing paper layer	5				5					
Cementing layer			40	30			40	30	40	30
Saturated felt layer, No. 1	15	15	15	15	15	15	15	15	15	15
Cementing layer			25	20			25	20	25	20
Saturated felt layer, No. 2	15	15	15	15	15	15	15	15	15	15
Cementing layer	25	25	25	20	25	25	25	20	25	20
Saturated felt layer, No. 3	15	15	15	15	15	15	15	15	15	15
Cementing layer	25	20	25	20	25	20	25	20		
Saturated felt layer, No. 4	15	15	15	15	15	15	15	15		
Cementing layer	25	20	25	20						
Saturated felt layer, No. 5	15	15	15	15						
Flood coat	70	60	70	60	70	60	70	60	70	60
Roofing slag	300	300	300	300	300	300	300	300	300	300
Or roofing gravel	400	400	400	400	400	400	400	400	400	400

* When laid over gypsum roof decks, use 15 lb. (2 gals.). See also[666].

the slag or gravel. An alternate procedure involves the use of rectangular pieces of roofing slate, laid side by side in a bed of coal-tar pitch or asphalt.[667] It has also been suggested that the usual slag or gravel surfacing be finished off with a comparatively thick layer of Portland cement, trowelled to a smooth finish, similar to sidewalk construction.[668] A well-known form of 5-ply slag or gravel built-up roof laid over wood and concrete, respectively, is illustrated in Fig. 188.

The following specifications have been formulated for surfacing materials:[669] Gravel or slag, or crushed stone shall be hard, dry, free from sand, dirt, clay, loam or other foreign substance.

Crushed stone shall be any desirable hard rock other than limestone. In any case they shall be of the following sizes—passing ¾-in. sieve 100 per cent; passing ⅝-in. sieve and retained on ¼-in. sieve, min. 80 per cent; retained on ⅛-in. sieve 100 per cent. A bituminous-concrete surfacing has been suggested for application over slag or gravel, to form a wearing course.[670] Promenade tile



Courtesy of The Barrett Co.

FIG. 188.—Tar-and-gravel Roof (5-ply over Wood and Concrete).

shall consist of vitrified shale tile of approved color and without visible defects, having a grooved back, and shall be of dimensions as may be specified. Slate shall have a minimum thickness of 3/16 in. with the sides cut true, and of a quality, size and color as may be specified.

Another type of built-up roof is finished with a so-called "cap-sheet" consisting of either smooth-surfaced or granular-surfaced roll-roofing, and without further treatment. Where granular-sur-

faced roll-roofing is used, it has been suggested that only half of the sheet in a longitudinal direction be surfaced with the granules,[671] or as an alternative that the granules be distributed longitudinally on the upper right hand and lower left hand halves of the sheet, respectively.[672] Another procedure consists in slotting, or partly punching holes through the sheets of saturated felt so as to form a better key with the adhesive material;[673] or in corrugating the sheet of saturated-felt and filling the corrugations with coating, so as to increase its thickness.[674]

Built-up roofs may be laid over metal roof decks, which are first insulated with a layer of "celotex,"[675] or a layer of cork blocks.[676] A method has also been proposed for laying built-up roof in the cold, with the use of plastic bituminous cement (i.e., "asphalt putty") or with bituminous emulsions in place of melted asphalt or coal-tar pitch.[677]

The following specification has been proposed for the so-called smooth roll-roofing surfaced with coarse talc:[678] minimum weight of dry felt used in its manufacture No. 38; minimum average weight per roll of 216 sq. ft. (without nails or lap-cement) 68 lb.

Laying Asphalt-shingle Roofs. These are customarily laid over solid wooden sheathing boards in the manner previously outlined. They may also be laid over old wooden shingle roofs, either directly, or with the use of tapered wooden filler strips inserted in the steps between the successive layers of the wooden shingles, so as to form a flush deck.[679] The following variations have also been suggested: laying the shingles in courses over strips of roll-roofing;[680] laying the shingles in two superimposed courses to accentuate their thickness;[681] lapping the sides of the shingles of successive courses in opposite directions;[682] laying the shingles over a sheeting printed with guide lines to facilitate their alignment and spacing;[683] using gaging strips properly to space the shingles;[684] using metal strips to secure the shingles at the roof edges;[685] etc.

Fire-resisting Properties of Prepared Roofings and Shingles. The Underwriters' Laboratories, Inc., of Chicago, Ill., established and maintained by the National Board of U. S. Fire Underwriters, classify fire-retardant roofings into three groups, as follows:[686]

CLASS A COVERINGS

Class A includes roof coverings which are effective against severe fire exposures. Under such exposures roof coverings of this class are not readily flammable and do not carry or communicate fire; afford a fairly high degree of heat insulation to the roof deck; do not slip from position; possess no flying brand hazard, and do not require frequent repairs in order to maintain their fire-resisting properties.

Built-Up

Asphalt Asbestos-felt Built-up Roof Coverings are composed of asphalt cement and asphalt-saturated asbestos-felt in the following combinations: (1) four layers of 14-lb. (18-lb. when coated) felt; (2) two layers of 28-lb. (each of 2-ply of 14-lb.) felt; (3) one layer of 35-lb. (55-lb. when coated) felt and two layers of 14-lb. (18-lb. when coated) felt; (4) three layers of 14-lb. (18-lb. when coated) felt, limited to noncombustible decks; (5) two layers of 35-lb. (55-lb. when coated) felt, limited to noncombustible decks. Built-up roof coverings of asbestos-felt are coated with asphalt, but need not be surfaced with roofing gravel, crushed stone or slag. Limited to roof decks having inclines not exceeding 12 in. to the foot horizontal when cemented and nailed to decks and inclines not exceeding 3 in. when cemented to the deck and not nailed.

Asphalt Rag-Felt Built-up Roof Coverings are composed of asphalt cement and asphalt-saturated rag-felt and/or combinations of cap and base sheets as listed below (minima specified), surfaced with approximately 400 lb. of roofing gravel or crushed stone or 300 lb. of crushed slag per 100 sq. ft. of finished roof. Limited to roof decks having inclines not exceeding 3 in. to the horizontal foot.

1. Four layers of 14-lb. felt.
2. One layer of 28-lb. and two layers of 14-lb. felt.
3. One layer of 14-lb. felt and two layers of cap or base sheets.
4. Three layers of cap or base sheets.
5. Three layers of 14-lb. felt. Limited to noncombustible roof decks.

Asphalt Asbestos-Felt and Rag-Felt Combination Built-up Roof Coverings are composed of asphalt cement, asphalt-saturated rag-

felt and asphalt-saturated asbestos-felt as listed below (minima specified) coated with asphalt, but need not be surfaced with roofing gravel, crushed stone, or slag. Limited to roof decks having inclines not exceeding 12 in. to the horizontal foot when cemented and nailed to decks, and to inclines not exceeding 3 in. when cemented to deck and not nailed.

1. One layer of 28-lb. rag-felt and two layers of 14-lb. (18-lb. when coated) asbestos-felt. Limited to noncombustible roof decks.

Tar Asbestos-Felt or Rag-Felt Built-up Roof Coverings are composed of coal-tar pitch and tar-saturated felt in combinations as listed below (minima specified), surfaced with approximately 400 lb. of roofing gravel or crushed stone, or 300 lb. of crushed slag per 100 sq. ft. of finished roof. Limited to roof decks having inclines not exceeding 3 in. to the horizontal foot.

1. Four layers of 14-lb. tar felt.
2. Three layers of 14-lb. felt. Limited to noncombustible roof decks.

Prepared

Asphalt Asbestos-Felt Sheet Roof Coverings are composed of sheets of 4-ply asphalt and asbestos materials laid in accordance with the instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines not exceeding 12 in. to the horizontal foot.

CLASS B COVERINGS

Class B includes roof coverings which are effective against moderate fire exposures. Under such exposures roof coverings of this class are not readily flammable and do not readily carry or communicate fire; afford a moderate degree of heat insulation to the roof deck; do not slip from position; possess no flying brand hazard; but may require infrequent repairs in order to maintain their fire-resisting properties.

Built-Up

Asphalt Asbestos-Felt Built-up Roof Coverings are composed of asphalt cement and asphalt-saturated asbestos-felt in combinations as listed below (minima specified) coated with asphalt but need not be surfaced with roofing gravel, crushed stone or slag. Limited to

roof decks having inclines not exceeding 12 in. to the horizontal foot when cemented and nailed to decks, and to inclines not exceeding 3 in. when cemented and not nailed.

1. Three layers of 14-lb. (18-lb. when coated) felt.
2. Two layers of 35-lb. (55-lb. when coated) felt.
3. One layer of 35-lb. (55-lb. when coated) felt, and one layer of 14-lb. (18-lb. when coated) felt.

Asphalt Rag-Felt Built-up Roof Coverings are composed of asphalt cement and asphalt-saturated rag-felt and/or combinations of cap and base sheets as listed below (minima specified), surfaced with approximately 400 lb. of roofing gravel or crushed stone or 300 lb. of crushed slag per 100 sq. ft. of finished roof. Limited to roof decks having inclines not exceeding 3 in. to the horizontal foot.

1. Three layers of 14-lb. felt.
2. Two layers of cap or base sheets.

Tar Asbestos-Felt or Rag-Felt Built-up Roof Coverings are composed of coal-tar pitch and tar-saturated felt as listed below (minima specified), surfaced with approximately 400 lb. of roofing gravel or crushed stone, or 300 lb. of crushed slag per 100 sq. ft. of finished roof. Limited to roof decks having inclines not exceeding 3 in. to the horizontal foot.

1. Three layers of 14-lb. felt.

Prepared

Asphalt Asbestos-Felt Sheet Roof Coverings are composed of sheets of 3-ply asphalt and asbestos materials, or asphalt asbestos-felt grit-surfaced, laid in accordance with the instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines not exceeding 12 in. to the horizontal foot.

Asphalt Asbestos-Felt Shingle Roof Coverings are composed of asphalt asbestos-felt grit-surfaced shingles, laid in accordance with instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines exceeding 4 in. to the horizontal foot.

Asphalt-Plastic Shingle Roof Coverings are composed of asphalt-plastic grit-surfaced shingles, laid in accordance with instruction sheets accompanying packages. Limited to roof decks capable

of receiving and retaining nails and to inclines exceeding 4 in. to the horizontal foot.

CLASS C COVERINGS

Class C includes roof coverings which are effective against light fire exposure. Under such exposures roof coverings of this class are not readily flammable and do not readily carry or communicate fire; afford at least a slight degree of heat insulation to the roof deck; do not slip from position; possess no flying brand hazard; and may require occasional repairs or renewals in order to maintain their fire-resisting properties.

Built-Up

Asphalt Rag-Felt Built-up Roof Coverings are composed of asphalt cement and asphalt-saturated rag-felt or combinations of cap and base sheets as listed below (minima specified). Limited to roof decks having inclines not exceeding 12 in. to the horizontal foot.

1. Three layers of 14-lb. felt.
2. One layer of 28-lb. and one layer of 14-lb. felt.
3. Two layers of cap or base sheets.
4. Two layers of 14-lb. felt and one layer of cap or base sheet.

Prepared

Asphalt Rag-Felt Sheet Roof Coverings are composed of rag-felt, either smooth or grit-surfaced, laid in accordance with the instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines exceeding 4 in. to the horizontal foot.

Asphalt Rag-Felt Shingle Roof Coverings are composed of asphalt rag-felt grit-surfaced shingles, one or more thicknesses, laid in accordance with instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines exceeding 4 in. to the horizontal foot.

Asphalt Asbestos-Felt Sheet Roof Coverings are composed of asphalt asbestos-felt, smooth surfaced, laid in accordance with instruction sheets accompanying packages. Limited to roof decks capable of receiving and retaining nails and to inclines exceeding 4 in. to the horizontal foot.

ROOFING CEMENTS AND SATURATED FELTS FOR BUILT-UP COVERINGS

Roofing cements and saturated felts, in certain cases with mineral surfacing (cap and base sheets), are used in the construction of built-up roof coverings.

These materials may be used to form Class A, B, or C built-up roof coverings depending upon the number of thicknesses of felt and whether mineral surfacing is used. Methods of assembling the various materials to form coverings of the respective classes are prescribed in the general information given above.

Additional fire-retardant tests have been conducted by the Bureau of Standards, Washington, D. C.,[687] and the Dominion Fire-Prevention Association, Ottawa, Canada.[688]

BITUMINIZED FLOOR COVERINGS

Saturated Felt Base. These first appeared on the market in 1910 as a substitute for linoleum, and have since enjoyed steadily increasing sale, due to their lower price, superior waterproof properties and remarkable resistance to wear and tear under foot. They are prepared from an asphalt-saturated rag-felt printed on the surface in colored patterns, manufactured with or without a backing of burlap, and in all cases faced on the underside with a suitable paint. When burlap is used, the asphalt-saturated felt may be regarded as replacing the central layer of oxidized linseed oil, resins, wood flour, powdered cork, colored pigments and mineral filler ordinarily employed in manufacturing printed linoleums. Where no burlap is used, the asphalt-saturated rag-felt may be viewed as the equivalent of both the central layer and burlap backing.

In manufacturing the floor covering, a sheet of rag-felt of high tensile strength, great uniformity in texture and thickness, is first saturated with an asphaltic mixture having a somewhat higher fusing-point and greater hardness than used for manufacturing roofing,[689] or a felt made of refined wood-pulp.[690]

At the present time No. 45 felt is used to make a saturated sheet weighing between 1¾ and 2 lb. per sq. yd., and No. 60 felt to make a saturated sheet weighing 2½ lb. per sq. yd. (unbacked and uncoated).

A colored bituminous saturant has been suggested composed of a mixture of wax tailings and colored mineral pigment; [691] or else dyeing the felt with an aniline dye and then saturating with a mixture of wax tailings and fatty-acid pitch; [692] or else the use of wax tailings hardened by steam-distillation, combined with a soluble dye or mixed mechanically with a colored mineral pigment, and emulsified with clay and water; [693] or else rosin oil hardened with manganese and lead resinate.[694]

The underside of the saturated sheet is backed with a colored paint composed of gloss oil, hardened rosin, colored pigment and solvent, by passing it through a rotary coater, whereupon it is festooned in ovens and allowed to bake for five to six hours at 120–140° F. When dry, the upper surface of the sheet is "primed" or "filled" with a mixture composed of heavy-boiled linseed oil ground with a filler, such as "talckene," and thinned to spreading consistency with a volatile petroleum distillate.[695] The function of the filler is to impart a so-called "linoleum" color to the mixture and cause it to dry with a semi-flat finish. The priming coat is also applied by passing the sheet through a rotary coating machine, whereupon it is festooned in ovens at 140 to 150° F. from six to twelve hours, until the priming coat has thoroughly dried.

Alternate procedures consist in applying the facing and backing coats of the solvent type to both sides of the sheet while it is still hot; [696] or an aqueous emulsion of the paint may be used for this purpose.[697] Sometimes the upper surface of the saturated felt is first sized with an aqueous solution of some glutinous substance, such as wheat flour, casein,[698] glue and silicate of soda,[699] an alkyd type of resin (e.g., glyptal),[700] or the like, with or without the admixture of a suitable filler to prevent the dark-colored saturant working its way through, and discoloring the paint subsequently applied to the surface. A similar paint wash may be applied to the underside of the sheet; [701] or the underside may be coated with a molten mixture of montan wax with colored mineral pigment,[702] or with a mixture of hardened rosin, paraffin wax and colored pigment; [703] or by applying a pyroxylin lacquer or other adhesive in which granulated cork is embedded; [704] or an aqueous emulsion containing rubber latex and mineral pigments; [705] or a paint containing metallic bronze powder mixed with ground mica; [706] or an aqueous-

oil emulsion and granulated cork;[707] etc. Another alternative consists in applying a backing composed of a compacted layer of masticated waste resulting from the manufacture of asphalt-impregnated felt-base floor coverings.[708] The use of a light-colored felt base consisting of bleached alpha-wood-pulp, printed in designs, and impregnated with a transparent lacquer, has also been suggested.[709]

Method of Printing. If the flooring is to be finished in designs to produce what is known to the trade as "print goods," it is next passed through a printing machine of the type ordinarily used for manufacturing linoleum, which will apply a number of colors in predetermined patterns or designs. These may be varied at will by changing the "print blocks" or dies used on the machine. The colored printing mixture contains suitable proportions of "gloss oil," mixed with colored pigments in quantities sufficient to color the mixture, but not to overcome the gloss when dry. Metallic bronze powder or metallic foil may be mixed with the coating.[710]

The printed goods are again festooned in the drying ovens at 140–180° F., where they are allowed to remain from 1 to 3 days until the surface coating dries hard.

In manufacturing "grained" floor coverings in imitation of wood, the sized and primed fabric is passed through a "Posselius" graining machine, which applies colored paint in the form of a graining to imitate the wood selected. This is allowed to dry and the surface finished with a coat of high-grade floor varnish, in some cases containing "gloss oil" when it is desired to increase the pliability of the finishing coat. The fabric is then suspended in the drying oven and heated until hard and tough.

The finished floor covering is marketed in the form of "piece goods" 74 in. wide in rolls 30 to 33 yd. long; also as rugs having a border on all four sides, measuring 9 by 12 ft. and smaller.[711] The durability of the product is said to be increased by the application to the surface of a cellulose-ester lacquer;[712] or by applying to the saturated felt a coating composed of cellulose-ester, rosin ester, a plasticizer, filler and colored pigment, with or without the addition of wax.[713]

A number of variations have been suggested from time to time, including the application to the asphalt-saturated felt base a layer of linoleum composition (oxidized linseed oil, resin, cork and pig-

ments);[714] or the application of untreated cloth fabric;[715] or applying an aqueous dispersion of asphalt to the underside of a needle-punched woven fabric;[716] or in using an asphalt-saturated felt with a pile fabric embedded in the upper coating;[717] or in using an asphalt-saturated felt with a layer of dry-felt cemented to the upper surface;[718] or in using an asphalt-saturated felt surfaced on both sides with asphalt in which sand is embedded;[719] or in applying granular mineral matter to an asphalt coated and saturated felt and buffing the surface to smooth same and expose the granules;[720] or in applying a surface-coating of asphalt provided with indentations;[721] or a surface-coating composed of a mixture of fatty-acid pitch, granulated cork or vegetable fibers and mineral filled;[722] or a surface-coating composed of asphalt emulsion, fillers and a mineral pigment;[723] or a surface-coating of plain asphalt followed by a second pigmented coating;[724] or surfacing the sheet with wood flour embedded in a cellulose-ester binder;[725] or in using a base having a sheet of unsaturated paper or felt embedded in the upper coating;[726] or one having a fabric embedded in the upper coating;[727] or in using for the base a laminated sheet, the lower portion of which consists of rag stock to which an emulsion of asphalt with clay and water is added in the process of manufacture, and the outer portion of which is composed of sized paper stock;[728] or in cementing together two layers of saturated felt with a relatively thick layer of bituminous matter in between;[729] etc.

To prevent the slippage of rugs, it has been proposed to indent or otherwise roughen the underside;[730] or by affixing a layer of unsaturated felt to the lower surface;[731] or by laying over a sheet of metal;[732] or by laying over concrete with a bituminous adhesive.[733] Various methods have also been patented by which the edges may be reinforced.[734]

WATERPROOFING MEMBRANES

The term "membrane" as applied to waterproofing was originally suggested by E. W. DeKnight, and alludes to a continuous sheet of bituminized fabric surrounding the structure to be waterproofed. The membrane system is also sometimes termed the "elastic" method, to distinguish it from the "integral" or "rigid" method of waterproofing. The former is ordinarily used for

waterproofing structures in the course of erection, and particularly the portion below ground level, including foundations of buildings, retaining walls, railway bridges, tunnels, subways, reservoirs, masonry tanks, swimming pools, etc. The fabric constituting the membrane may be felted or woven, or a combination of the two.

Materials Used. The materials ordinarily used for this purpose include the following:

Fabrics:

(1) Tarred felt.
(2) Asphalt-saturated felt.
(3) Raw burlap or duck.
(4) Tarred burlap or duck.
(5) Asphalt-saturated burlap or duck.
(6) Asphalt-saturated and coated felt surfaced with fine mineral matter, moderately coarse embedded mineral matter, wood-flour or sawdust; also asphalt-saturated felt, coated on one side with asphalt.[735]
(7) A laminated sheet of bituminated fabric composed of bituminized felt, burlap, drill,[736] or duck, used alone or in various combinations.
(8) Sheet metal cemented with asphalt plastic to bituminized fabric.[737]
(9) Sheet glass cemented with asphalt plastic to bituminized fabric.[738]

Open-mesh fabrics are recommended, so that the ply-cement may form a better "key." For this purpose, it is proposed to coat the fabric with asphalt and then blowing air against same before the asphalt has cooled so as to keep the meshes from being scaled.[739]

Bituminous Adhesive Compounds:

(1) Coal-tar pitch or mixtures of coal-tar pitch with water-gas tar pitch.
(2) Asphaltic compounds.

The weight of felted fabrics used for waterproofing purposes varies widely, but the best practice provides that the dry felt shall not be less than No. 30 on the felt makers' scale (6.25 lb. per 100 sq. ft.) and the bituminized felt not less than 14 lb. per 100 sq. ft. Saturated and coated felts are also made in various weights ranging from 14 lb. to 70 lb. per 100 sq. ft.

Of the woven fabrics, burlap is generally employed, varying in weight from 10 to 20 oz. per square yard after saturation. The use of a cotton fabric is advocated instead of burlap, since cotton is less liable to rot on continuous contact with moisture, but on the other hand it is more expensive. The cotton, burlap or felt may be made more resistant to decay by impregnation with a solution of copper or iron sulfate;[740] organic copper compounds;[741] "pitch oil" and boracic acid;[742] mercuric chloride;[743] red oxide of mercury;[744] zinc chloride;[745] zinc arsenite with creosote;[746] arsenious acid with ferric hydroxide;[747] arsenious oxide;[748] arsenious oxide with potassium dichromate;[749] zinc borate with fluorine, anthracene, phenanthrene, carbazole, or the like, and a chlorinated tar acid.[750] The fabric upon being treated with chemicals must be dried before being waterproofed with asphalt or coal-tar pitch. One procedure consists in first saturating the treated fabric with melted paraffin wax, followed by saturation with asphalt.[751] The finished product should carry from 60 to 66⅔ per cent bituminous matter, and the chemicals act as preservatives and prevent the fibers from mildewing.

No definite statements can be made regarding laminated fabrics composed of felt and burlap, since these vary widely in their structure and weights.

There has been much discussion whether coal tar or asphaltic products are superior for waterproofing masonry.[752] The present practice seems to favor the use of asphaltic products when they are to be subjected to air, sunlight or vibration, and the use of coal-tar products where the waterproofing is protected from these agencies. This conforms with the author's experience.

The specifications for the adhesive compounds have already been given in Tables LII and LIII. The priming coat for use in conjunction with a mopping of hot coal-tar pitch shall comply with the following specifications:[753]

The creosote oil shall be a pure tar distillate free from any substance foreign thereto; it shall be entirely fluid at 100° F.; its specific gravity at 100° F./77° F. shall not be less than 1.00 nor more than 1.06; it shall show not more than 1 per cent insoluble in hot benzol; when distilled it shall yield not exceeding 2 per cent of water, not exceeding 5 per cent by weight distilling under 200° C., not exceeding 50 per cent nor less than 30 per cent distilling under

235° C., and not exceeding 15 per cent of residue at 355° C., which must be soft in consistency at 77° F. The specific gravity at 100° F./77° F. of the fraction distilling between 235 and 315° C. shall not be less than 1.00.

Similarly, the specifications [754] for the primer to be used in conjunction with asphaltic adhesives, are as follows:

The primer shall consist of a paint containing 25 to 35 per cent by weight of asphaltic base complying in every respect with the asphaltic adhesive compound, thinned to ordinary paint consistency with a hydrocarboon distillate having an end-point on distillation of not above 260° C., of which not more than 20 per cent shall distil under 120° C. The primer shall be free from water and contain not more than 1 per cent sediment.

Preparing the Underlying Surface. To insure the courses of bituminized fabric adhering properly to the underlying surface and to each other, the following precautions should be observed:

(1) Where the membrane is to be applied below grade, an adequate drainage system must be provided so that the masonry surface will be thoroughly *dry* during the installation of the waterproofing.

(2) It is of the utmost importance that the work should not be undertaken in rainy, snowy or very cold weather.

(3) Concrete surfaces should be brushed, scraped or chipped to remove all sharp projections which would puncture the waterproofing; also any dirt, foreign matter or cement which may have been raised to the surface during the placing of the concrete.

(4) Masonry surfaces should be primed with creosote oil when coal-tar products are to be used, or with an asphaltic paint when asphaltic products are to be used for waterproofing.

(5) Metal surfaces should be cleaned to remove all rust, scale, dirt or grease, and primed with a paint containing either coal-tar pitch or an asphaltic base, depending upon the character of waterproofing used.

(6) The floors of steel railroad bridges should first be covered with a 1 : 3 : 5 concrete containing ¾ in. stone or gravel, the surface of which when set and dry should be treated as specified in (3).

(7) When coated fabrics are used, the adhesion may be promoted by omitting the coating from one side of the saturated felt,[755] since the melted bituminous adhesive forms a better bond with plain saturated felt than felt surfaced with a bituminous coating of harder consistency.

(8) Saturated and coated felts may be surfaced with wood flour, fine sand, or sawdust, instead of talc, soapstone or other finely divided mineral filler, because the mineral powders exert a repellant action on the melted bituminous adhesive.

(9) When burlap is used in a single sheet, it is customary to manufacture it with the bituminous coating applied in such a manner that the meshes between the strands will remain open, the theory being that the melted adhesive will fill these and thus key itself more securely to the bituminized fabric.

(10) *Not less than 25 lb. of coal-tar pitch or asphaltic adhesive shall be applied per 100 sq. ft. of underlying fabric or masonry.*

The use of an adhesive (tacky) asphalt paint has been proposed for cementing asphalt-coated and saturated sheets of felt in the cold, without recourse to heat.[756]

Selecting and Installing the Waterproofing Membrane. Whether a felted or a woven fabric or a combination of the two is to be used, depends upon the character of the work and the preference of the engineer in charge. A woven fabric is more pliable, stronger, and less liable to tear or break when bent over sharp corners, but to counterbalance these, the felted fabric is less expensive, more durable and more resistant to moisture. Modern practice accordingly favors a combination of the two.

The number of courses of bituminized fabric to be applied depends upon the head of water encountered, the factor of safety sought and the views of the engineer in charge of the work. There is no standard practice followed in this connection. Where tarred or asphalt-saturated felt weighing 14 lb. per 100 sq. ft. is used, the figures in Table LXXV will serve as a safe guide.

The fabrics should *always* be laid "shingle fashion," and *all* the layers applied in the *same* direction.

(*a*) When the membrane is to be composed of two courses, lay the fabric shingle fashion, lapping each course 1 in. more than half the width of the preceding one.

(*b*) When the membrane is to be composed of three courses, lay the fabric shingle fashion, lapping each course 1 in. more than one-third the width of the preceding one.

(*c*) When the membrane is to be composed of four courses, first cover the surface with two layers of fabric laid shingle fashion, lapping each course 1 in. more than half the width of the preceding

TABLE LXXV
HYDROSTATIC PRESSURES

Hydrostatic Head, Feet	Pressure in Pounds per Square Foot Under Floor	Average Pressure in Pounds per Square Foot Against Side Walls	Number of Courses Tarred or Asphalt-Saturated Felt to be Used
0	0 0	0 0	2
1	62.5	31.2	3
2	125.0	62.5	4
6	375.0	187.5	5
8	500.0	250.0	6
10	625.0	312.5	7
15	937.5	468.7	8
20	1250.0	625.0	9

one, and then follow with two additional courses installed in the same manner.

(*d*) When the membrane is to be composed of five courses, follow the procedures indicated in (*b*) and (*a*) respectively.

(*e*) When the finished membrane is to be composed of six courses, duplicate the procedure outlined in (*b*).

In the case of railroad bridges, where the conditions are extremely severe, the following alternative specifications are recommended:[757]

(1) From four to six courses of bituminized felt.
(2) A middle course of bituminized duck with two courses of bituminized felt on either side.
(3) A bottom course of bituminized felt, followed by two courses of bituminized duck with two upper courses of bituminized felt.
(4) Two or three courses of bituminized duck.
(5) Combinations of the foregoing with asphalt mastic.

It is extremely important that the work should be *continuous*. A lack of continuity will be fatal to any membrane, since the water is sure to find its way through. Each layer of pitch, asphalt or other adhesive should *completely* cover the surface over which it is spread, without breaks, blow-holes or other imperfections. It has been proposed that the melted adhesive be pumped through a hose under pressure and conveyed directly to the surface where it is to be applied.[758] The fabric must be rolled out smoothly, and pressed

into the hot cementing material, to insure its sticking thoroughly and evenly. Where it becomes necessary temporarily to discontinue the work, laps at least 12 in. wide must be provided to join with the ensuing section of the waterproofing. On walls connecting with floors, the ends of the floor layer should be carried through the wall and turned upwards outside, whereupon the fabric on the outside of the walls should be carried down over the ends of the floor layer, lapping at least 12, and preferably 24 in. On connecting the wall with the roof work, the layers of fabric from the roof should be carried down over the outside of the wall layers forming a lap at least 12, and preferably 24 in.

Protecting the Waterproofing Membrane. After installing the membrane, it must be protected against any mechanical injury which is liable to occur on backfilling with earth, depositing concrete, or bricking in; also against sagging, bulging, or running, when subjected to intense summer heat. The following means are adopted for this purpose:

(1) On walls it is customary to protect the membrane with brickwork, or a facing of 1 : 3¾ Portland-cement mortar ⅜ to ½ in. thick.

(2) On flat surfaces, the membrane is covered with concrete or bricks embedded in cement grout or a bituminous grout.

(3) Where the membrane is laid on level floors of railroad bridges, very good results have been obtained by covering it with a 1½-in. layer of asphalt mastic flooring, which is sometimes covered by a course of asphalt-saturated felt.[759]

Table LXXVI gives a résumé of the damp-proofing and waterproofing methods applicable under different conditions to various structures.[760]

INSULATING AND BUILDING PAPERS

Insulating and building papers are manufactured from special paper stock waterproofed with bituminous mixtures in one of three different ways, and according to which the following classes are recognized,[761] viz.:

(1) Coated but not saturated.
(2) Saturated but not coated.
(3) Both saturated and coated with bituminous compositions.

The function of the insulating or building paper is to prevent the transfer of heat from the outside to the inside of a closed chamber or building. Expressed in another way, its purpose is to keep the inside either cooler or warmer than the surrounding atmosphere. In the case of cold-storage plants, railroad refrigerator cars, ice chests or vaults, insulating papers are used to keep the interior *cool;* whereas with buildings and residences heated artificially in winter, the function of the building or sheathing paper is to keep the interior *warm,* by preventing the egress of heat.

Waterproof papers are also used for temporarily covering freshly laid cement pavements and roadways to prevent the premature evaporation of water under the heat of the sun, also freezing at low temperatures, thereby promoting the setting of the cement.[762]

Insulating and sheathing papers are also used to line walls, floors, and sometimes ceilings in one or more layers; also to cover the exterior of brick walls before applying stucco.[763] The greater the number of layers used, the more efficient will be the installation. For buildings and residences, usually one layer of the paper is employed, whereas for cold-storage plants, refrigerator cars, and ice chests, it is customary to use two or more layers of the paper with air spaces in between. The least expensive and most efficient form of insulation consists in confining a "dead" or non-circulating air space between two or more layers of insulating paper.

In practice, the paper is introduced in the floors, walls and partitions of buildings between protective layers of wooden boards. In other cases, the insulation is manufactured in the form of flat sheets composed of vegetable or animal fibers (e.g., flax, hair, etc.) sewn or otherwise fastened between two layers of bituminized paper, and known to the trade as "flax felt," "hair felt," etc. Rag-felt coated superficially with asphalt has been used for heat-insulation and sound-deadening purposes.[764]

Untreated paper answers poorly for insulating purposes, as it will become affected by the moisture and dampness, resulting from the condensation which occurs whenever a cool surface comes in contact with warmer air carrying a larger percentage of moisture. Under the influence of moisture, raw paper soon disintegrates and loses its value as an insulator. It is necessary, therefore, to water-

TABLE LXXVI

DESCRIPTION OF WATERPROOFING AND DAMP-PROOFING METHODS*

No.	Materials Employed	Method of Use	Thickness	Remarks
	Surface Coatings.			
A	Clear damp-proofing paints.	Brushed cold on exterior surfaces.	2 coats	For damp-proofing new or old work above ground, also to preserve building stone and prevent efflorescence.
B	Paraffin melted and applied hot.	Brushed hot on exterior surfaces previously warmed.	Penetrates $\frac{1}{4}$ to $\frac{1}{2}$ in.	
C	Black damp-proofing paints and bituminous emulsions.	Brushed cold on interior surfaces.	2 coats.	Used to replace furring and lathing.
D	Bituminous cements.	Trowelled cold on exterior or interior surfaces.	1 coat, $\frac{1}{16}$ to $\frac{1}{8}$ in. thick.	Used for same purposes as black damp-proofing paints, also to a limited extent on outside of walls.
E	Bituminous adhesive compounds.	Mopped hot on exterior surfaces.	1 mopping, $\frac{1}{16}$ to $\frac{1}{8}$ in. thick.	Used on outside of walls where conditions are not severe.
	Integral methods:			
F	Cement-mortar with waterproofing compound.	As a facing on interior surfaces exposed to moderate or great heads of water; also as a stucco on exterior walls.	$\frac{3}{4}$ to 1 in. on floors; $\frac{1}{2}$ to $\frac{3}{4}$ in. on walls.	Used as facings on new work or to remedy defective old work; also as stucco on new work.
G	Carefully graded concrete with waterproofing compound.	Mixed in the body or mass of the concrete when formed.	Throughout the concrete.	Suitable only for new work in the course of construction.
	Membrane method:			
H	Bituminized fabrics employed in conjunction with bituminous adhesive compounds.	In alternate layers on exterior surfaces exposed to a head of water.	2 to 10 layers.	Suitable only for subgrade work in the course of construction.
	Plastic method:			
I	Asphalt mastic.	As a plaster or coating on surfaces exposed to moderate heads of water.	$\frac{1}{2}$ to 1 in. thick.	Gives the best results on horizontal surfaces in new or old work; difficult to apply on vertical surfaces.
	Membrane and plastic method:			
J	Bituminized fabrics employed in conjunction with asphalt mastic.	In alternate layers on surfaces exposed to moderate or great heads of water.	2 to 5 layers of fabric with a total of $\frac{1}{4}$ to 1 in. of mastic.	Suitable only for new work. Capable of withstanding vibration.

* *Waterproofing* (noun): A treatment to prevent the passage of water under pressure.
Damp-proofing (noun): A treatment to resist the passage of moisture in the absence of pressure.

proof the raw paper to enable it satisfactorily to resist decay. Asphaltic materials and waxes are ordinarily employed for this purpose.

Raw Paper Stock. A strong paper of open texture should be used for manufacturing insulating and sheathing papers. In accordance with the paper-makers' scale the raw stock [765] is designated by a "number," corresponding to the weight in pounds per ream

TABLE LXXVI—*Continued*

METHODS SUITABLE FOR VARIOUS STRUCTURES

Nature of Work	During Construction (New Work)	After Construction (Old Work)	Remarks
Buildings:			
Exposed walls: On inside	C, D	C, D	For damp-proofing walls and replacing furring and lathing.
Exposed walls: On outside	A, B	A, B	For damp-proofing and preserving stone and to prevent efflorescence.
Exposed walls: On outside	F	A	For waterproofing stucco and preventing cracks.
Exposed walls: On outside	D, E	D, E	For damp-proofing where appearance is immaterial.
Superstructural floors (in bathrooms, toilets, lavatories, stables, garages, etc.), swimming pools and tanks indoors.	F, H, I	F. I	For preventing seepage or else to retain water.
Foundation pits, wells, trenches, footings, basement walls and floors.	F, H	F	For resisting external water pressure.
Concrete blocks	A, F	A	For damp-proofing and preventing cracks.
Railroad structures:			
Subways, tunnels, arches, culverts, and retaining walls.	F, G, H	F	For resisting water pressure or preventing seepage, and in certain cases to withstand vibration (except F). Great skill required to waterproof *after* construction.
Bridges	H, I, J	F	
Water and sewage systems:			
Reservoirs	F, G, H, I	F, I	For resisting water pressure. Mastic (I) difficult to install when masonry once saturated with water.
Dams, conduits, filtering chambers, aqueducts, sewage disposal systems and manholes.	E, F, G	F	For preventing seepage of water or sewage through masonry.
Marine work:			
Concrete piles, piers, sea walls, and cement ships.	F, G	F	For protection against frost and the destructive action of sea water.

composed of 500 sheets, each measuring 2 by 3 ft.; in other words, by the weight in pounds of 3000 sq. ft. Raw papers from No. 30 to No. 180 are used, composed of one or more of the following classes of fibers, viz.:

(1) Jute, hemp and manila fibers.
(2) Chemical wood fibers (sulfite and sulfate fibers).
(3) Mechanical wood fibers (i.e., ground wood).
(4) Rag fibers (cotton and wool).

The first three classes are ordinarily used, and rag fibers in rare cases to open up the "texture" of the sheet, and enable it to saturate more readily.[766] Rags serve to increase the "thickness factor"

and correspondingly decrease the "strength factor" of the paper.

Jute, hemp and manila fibers impart toughness, strength and permanence to the paper. Chemical wood fibers also tend to strengthen the paper, but are not regarded with as much favor as the foregoing, since it is difficult and in some cases impossible to remove the last traces of chemicals, which in time are apt to decompose and weaken the paper. Chemical wood fibers are divided into two classes, viz.: sulfite and sulfate fibers respectively. The sulfate fibers are present in "kraft" papers [767] and represent one of the strongest of all fibers. Mechanical wood fibers are the first to "rot" and decrease in strength, and their proportion should therefore be kept as low as possible.

In ascertaining the value of the raw paper for insulating and sheathing purposes, preference should be given to stock carrying the smallest percentage of mechanical wood. It is advisable that this should not exceed 25 per cent of the total. The balance may consist of jute, hemp, manila and chemical wood fibers in varying proportions, or chemical wood fibers alone. A good kraft paper will consist of 100 per cent chemical wood (sulfate) fibers.

The paper should be loose in texture, showing a thickness factor* of 0.090 to 0.125, a strength factor† (ascertained by the Mullen tester, Test 79) ranging from 0.35 to 1.00 (averaging about 0.60), and not exceeding 3 per cent ash on ignition. The highest strength factors are shown by kraft papers which are sometimes guaranteed to test a pound on the Mullen tester, for each pound in weight on the paper makers' scale.

Bituminous Saturation. For saturating the paper, the bituminous composition may consist of the following: [768]

(1) Dark-colored mixtures similar to those used for saturating prepared roofings,[769] including blown asphalt, scale wax and montan wax.[770]

(2) Light-colored mixtures [771] containing one or more of the following products: paraffin wax; [772] ozokerite; [773] paraffin wax and rosin; [774] carnauba wax and rosin; [775] carnauba wax and wax tailings; [776] candelilla wax alone, or in admixture with beeswax and paraffin; [777] spermaceti; stearin; beeswax, Japan wax, guttapercha; [778]

* i.e. the thickness in mils divided by the "number" of the paper.
† i.e. the strength in pounds divided by the "number" of the paper.

mineral wax and viscous mineral oil;[779] petrolatum and rosin;[780] wax tailings and rosin,[781] etc. Light-colored waxes are used to saturate paper products in the manufacture of so-called "paraffined paper," also to saturate cloth for use as bandages, wrapping corpses (known as cerecloth), etc.

The life of the paraffin wax bath used for impregnating purposes may be prolonged by adding a trace (0.05 to 0.50%) of β-naphthol, pyrogallol, diphenylamine, gallic acid, hydroquinone, or *p*-phenylenediamine, which serve as anti-oxidants.[782]

An alternate procedure consists in grinding, or introducing an aqueous dispersion of wax with the paper fibers in the beater, prior to the formation of the web of paper on the paper-machine.[783] The following mixtures have been proposed for these purposes: a soap emulsion of paraffin wax and petrolatum;[784] a soap emulsion of paraffin wax containing precipitated magnesium carbonate (obtained by adding $MgCl_2$ to Na_2CO_3 solution) thereby making possible a higher wax concentration;[785] a soap emulsion of paraffin wax containing naphtha;[786] an emulsion of paraffin wax with a metallic salt of montan wax;[787] a soap emulsion of paraffin wax and viscose;[788] a soap emulsion of petrolatum;[789] an emulsion of petroleum resin (obtained from gas-oil resinification) with alkali;[790] an emulsion of chloronaphthalene followed by adding alum;[791] an emulsion of carnauba wax with trihydroxylethylamine and linoleic acid;[792] incorporating powdered montan wax with the aqueous fibrous stock in the beater;[793] etc.

A process has also been described involving the electro-deposition of wax on a web of paper from a colloidal wax dispersion.[794]

Bituminous Coating Compositions. Dark-colored asphaltic mixtures or hard coal-tar pitch[795] are used, similar to the coatings of prepared roofings, with the exception that they are usually manufactured harder in consistency and of a higher fusing-point. A small percentage of mineral, animal or vegetable wax (5 to 15 per cent) is sometimes added to impart wax-like properties and an "unctuous" feel.[796] Residual asphalts have also been proposed for coating purposes;[797] likewise a mixture of paraffin wax and rubber (added in the form of latex, or in solution and evaporated dry);[798] likewise a flame-retarding coating consisting of fatty-acid pitch surfaced with paraffin wax.[799]

Method of Manufacture. The raw paper is saturated, coated, or both saturated and coated by a machine similar to that used for manufacturing prepared roofings.[800] The web is saturated by running it through a tank of melted saturating material, usually heated by steam to a temperature of 225 to 350° F.[801] As paper stock is considerably denser than roofing felt (the thickness factor amounting to 1/7 to 1/8 of the latter) it will carry a correspondingly smaller percentage by weight of saturation. A well-saturated paper should contain not less than 33⅓ per cent of bituminous saturation based on its finished weight. The greater the percentage of saturation present, the more moisture-resistant will the paper be. In rare cases the saturated paper will carry as high as 50 per cent by weight of bituminous saturation. Another procedure consists in applying the bituminous matter to one side of the sheet, so that it will be absorbed into the paper and obviate a sticky surface.[802] Paper may be impregnated with melted asphalt at a temperature sufficient to char the fibers and thereby increase the rigidity of the product.[803]

A thinner coating is applied to the paper than in the case of prepared roofings. It will range from 10 to 25 lb. per 1000 sq. ft. (referring to the coating on *both* sides of the sheet), when applied to saturated papers, with an increase of about 25 per cent for papers which are coated but not saturated.

Insulating and sheathing papers are sold in rolls containing 1000, 500 and in some cases 250 sq. ft. There are no standard weights recognized by the trade, each manufacturer following his own views. Papers are marketed at the present time from 20–125 lb. per 1000 sq. ft., the heavy weights falling on the border-line between papers and "felts," often containing a proportion of rag stock.

Saturated and coated papers are more waterproof, more durable and better insulators than the other types. Similarly, a paper which is saturated is more efficient than one which is simply coated. The several methods of waterproofing are designed to meet the prevailing views of engineers, some preferring one and some another. Sometimes the purpose for which the paper is to be employed will predetermine the type used; for example, for surfacing sheets of fibrous material (flax-felt or hair-felt), saturated papers only

can be employed, since coated papers would gum up the needle of the machine used for sewing the sheets together.

The thicker the paper, the stronger and more durable it will prove to be. However, from the standpoint of insulation it is better to use two layers of a thin paper with an air space in between, than a single layer of thick paper equal to their combined thickness.

A base for manufacturing imitation leather has been proposed, consisting of an open-textured kraft paper impregnated with a mixture of asphalt and bodied linseed oil, and surfaced with a pyroxylin composition.[804]

BITUMINIZED PAPERS FOR ELECTRICAL INSULATION

Sometimes papers saturated and coated with asphalt or fatty-acid pitch [805] are used for electrical insulating purposes, as for example in constructing automatic telephone switchboards, for wrapping wires and cables, etc. The following tests were obtained by the author upon subjecting papers of this character to an alternating current increased at the rate of 125 volts per minute between flat disc terminals with rounded edges, the areas of contact measuring exactly 1 sq. in.:

	A	*B*	*C*	*D*
Weight in pounds per 1000 sq. ft. (Test 65*a*)	21	42	60	90
Thickness in mils. (Test 66)	5	8	12	20
Mullen strength at 77° F. (Test 67*b*)	25.0	30.0	40.2	72.2
Breakdown voltage at 77° F. (Test 72)	750	1190	1637	2650
Volts per mil	150	149	137	132

The insulating properties of the saturant may be improved by fluxing the asphalt with metallic salts of naphthenic acid (e.g., Ca, Ba, Al, Mg, Pb, Mn, Co or Fe naphthenate).[806]

BITUMINIZED PAPERS FOR WRAPPING AND PACKING PURPOSES

These include papers similar to the foregoing, likewise kraft paper stock either coated on one side only (which may be used as a sealing strip),[807] or duplexed by cementing two or more layers of paper with asphalt.[808]

Bituminized papers may be creped;[809] or combined with cellophane or aluminium foil;[810] or reinforced by combining with open-mesh fabrics or threads or strings.[811] An aqueous emulsion of asphalt has been suggested either for coating the raw paper,[812] or for sticking together two layers.[813] Corrugated papers have also been recommended for this purpose, assembled in various ways.[814]

BITUMINIZED PAPERS FOR MULCHING PLANTS AND CROPS

Excellent results have been obtained, particularly in pineapple culture on the island of Hawaii, through the use of a paper saturated, or saturated and coated with asphalt.[815] The plants are

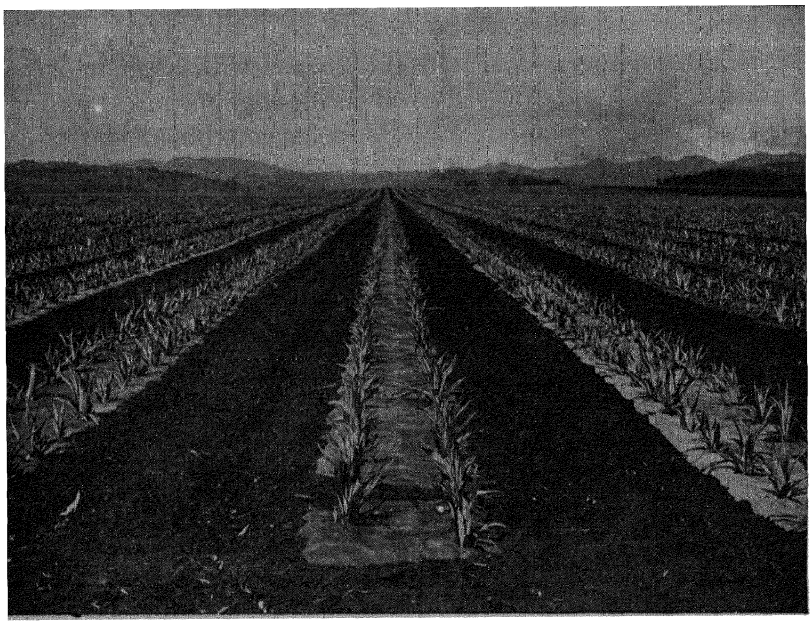


FIG. 189.—Use of Mulching Paper for Growing Pineapples.

planted through the paper, which is unrolled on the ground, as illustrated in Fig. 189. In 1933 about 30,000 acres were mulched in this manner. The paper accomplishes the following results, viz.: it checks the growth of weeds, it protects the soil from the compacting action and erosive effect of torrential rains, it conserves moisture during dry spells, and it increases the temperature of the soil because the black color of the paper absorbs more heat than the sur-

rounding soil. As a result, there is obtained an increased yield of pineapples ranging from 25 to 55 per cent. It is also claimed that the growth of other crops, such as tobacco, sugar-cane, vegetables, etc., may be similarly enhanced.[816]

BLASTING FUSES

These are illustrated in Fig. 190, and consist of twisted cotton strands *A* enclosing a core of gun-powder, water-proofed on the surface or "struck in" by being drawn through a die which applies

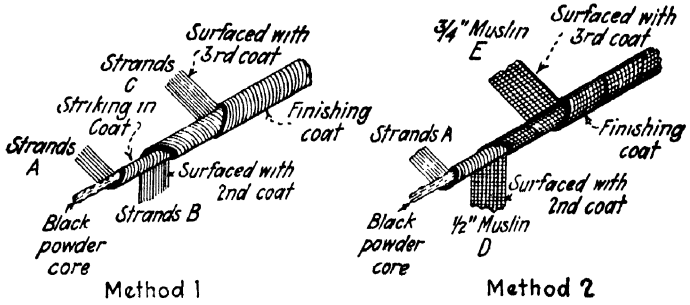


FIG. 190.—Blasting Fuses.

a soft bituminous composition melted at about 250° F. This should have a fusing-point of 140–150° F. (R. and B. method) and consists of blown petroleum asphalt, or fatty-acid pitch, or else steam-distilled residual asphalt (e. g., California D grade). The fuse may be finished in one of two ways: Method No. 1 consists in next wrapping the fuse with cotton strands *B*, surfaced just prior to their coming in contact with the fuse with a second coat of melted bituminous material, whereupon it is wound spirally in a reverse direction with strands *C*, similarly surfaced with a third coating. The fuse is then drawn through a larger die which applies a "finishing coat" of melted bituminous composition. Method No. 2 consists in applying two courses of muslin tape *D* (½ in. wide) and *E* (¾ in. wide) in a similar manner. In both methods the second, third and finishing coats are applied at 240–275° F. These coatings usually have similar compositions and show a fusing-point of 180–200° F. (R. and B. method). Fatty-acid pitch [817] tempered with gilsonite has been found to give the best results, since it becomes tougher in time, without growing brittle.

A mixture of fatty-acid pitch and mica has also been suggested.[818] Grahamite fluxed with Mexican residual oil or blown asphalt may also be used for the second, third and finishing coats, but is not as satisfactory. The surface of the fuse is finally coated with a mixture of white clay and water and wound into 50-ft. coils. The finished fuse measures 5/16 in. in diameter. The bituminous material waterproofs the fuse and enables it to be used in damp or wet localities without deterioration.[819]

BITUMINIZED CORDS AND ROPES

Cords and ropes may be weatherproofed by impregnating with petroleum asphalt,[820] wood tar, or soft coal-tar pitch, and are used for calking the joints on ships, pipe lines, etc.

INSULATION FOR ELECTRICAL TRANSMISSION WIRES

The cotton covering is either saturated with a bituminous mixture, or both saturated and coated therewith.[821] The saturation is similar to that used for impregnating prepared roofings. Asphaltic products of a harder consistency, corresponding to the weather-coatings of prepared roofings, are employed for coating cotton-covered wires exposed out-of-doors, such as electric light wire, known as "weather-proof insulated wire." Another type consists in covering the copper with a rubber composition having high electrical insulating properties, which in turn is "weather-proofed" by two or more protective layers of cotton braiding impregnated at 360 to 375° F. with a bituminous composition (e.g., gilsonite, blown asphalt and a small percentage of animal, vegetable or mineral wax). After cooling, the wire is then passed through a second tank of the "polishing" or "finishing" mixture, consisting of bituminous matter with a higher proportion of wax content, maintained at 300° F. When cool, the wire is finally passed through a mechanical contrivance, known as a "polisher." Another saturant of high dielectric strength consists of mineral oil (e.g., cylinder oil stock) heated with rubber latex and ethylene-diphenyldiamine until the water is expelled and the solution is complete.[822]

Bare copper wires may be insulated with certain varieties of fatty-acid pitch, which are transferred into an insoluble and infusible coating by heating to a high temperature,[823] or a coating of this

character may be encased in a layer of rubber.[824] Flame-resistant coverings may be produced by coating with fatty-acid pitch followed by a coating of cellulose-acetate lacquer,[825] or by the addition of selenium to the finishing mixture.[826] Wrappings of asphalt-saturated paper combined with cellophane have also been proposed;[827] likewise a coating of pigmented fatty-acid pitch surfaced with β-naphthol to inhibit molds and impart a non-sticky finish;[828] also a mixture of fatty-acid pitch, synthetic resin (e.g., phenol-formaldehyde or cresol-formaldehyde type) and castor oil.[829]

BITUMINIZED FIBER CONDUITS

These are used for protecting electrical transmission wires when housed in floors, walls and other inaccessible parts of buildings, and consist: (1) of solid tubes composed of fiber or other bibulous substance, impregnated and coated with asphalt or coal-tar pitch[830] or wax; or (2) bituminized paper or cardboard wound spirally in tube form and cemented together with bituminous matter.[831] To promote the adhesion of the asphalt coating to a fiber conduit impregnated with coal-tar pitch, it is suggested that a thin layer of fatty-acid pitch be applied between the two.[832] Other variations consist in using a mixture of blown asphalt, scale wax and montan wax for impregnation;[833] or in surfacing the conduit with paraffin wax, carnauba wax, montan wax, or the like;[834] or in introducing a metal foil in between the windings.[835] Containers may also be made of paper wound in concentric layers cemented together with asphalt.[836]

ASPHALT PIPES

These were first introduced in 1850 by M. Chameroi in France and by the Earl of Dundonald in England and used for water-pipes, etc. They consisted of asphalt-saturated paper wound on a cylinder in concentric layers, cemented together with melted asphalt.[837]

BRAKE LININGS AND CLUTCH-FACINGS FOR AUTOMOBILES

Asbestos rings are impregnated with a mixture of residual oil from Mexican petroleum thinned with gas oil and containing 3 to 5 per cent of Pb_3O_4. These are then baked at a slowly increasing

temperature, starting at 300° F. and terminating at 500° F., in twelve to eighteen hours. The heat is then increased to 550° F. and maintained for twenty-four to forty-eight hours.[838] In this treatment the asphalt is polymerized to a point where it is rendered insoluble in carbon tetrachloride or kerosene. The use of gilsonite and china wood oil has also been recommended for this purpose, in much the same manner;[839] likewise a mixture of gilsonite and hydrogenated china wood oil;[840] a mixture of asphalt and phenolic resin;[841] etc. Other alternatives consist in adding an asphalt emulsion to the asbestos fibers and water in a beater and forming the sheet in the conventional manner;[842] or grinding hard asphalt (e.g., gilsonite) with the asbestos fibers and water in the beater, and after forming the sheet, baking same to flux the asphalt,[843] or by first impregnating the sheet of asbestos with gilsonite or hard coal-tar pitch and sulfur, followed by heating and finally impregnating with a blown asphalt.[844] An analogous process consists in impregnating canvas machine belts with asphalt.[845]

INSULATING AND ACOUSTICAL FELTS FOR AUTOMOBILES

The steel tops and bodies of automobiles are insulated and sound-proofed by cementing an indented asphalt-saturated felt (which may be single-layered or laminated) within the steel body.[846] Sheets of felt partially impregnated with asphalt,[847] or bituminized felt in combination with aluminium foil [848] have also been recommended for this purpose.

Shim-strips have been used to prevent the squeaking of the component parts of automobile frames where riveted together, consisting of prepared roofing cut into narrow strips.[849]

Socalled "tacking-strips" consisting of ribbons of paper saturated with asphalt and twisted in the form of a rope, have been used for tacking the upholstery in place within the bodies of automobiles.[850]

ELECTRICAL INSULATING TAPE

Bituminized tapes for electrical insulating purposes are prepared by passing strips of cotton or muslin through a bath of melted bituminous material intended to fill the pores of the fabric and provide

a slight excess on the surface. They are manufactured in widths of ½, ¾, and 1 in. respectively, and wound in ¼ to ½-lb. rolls, averaging ⅛ lb. The length per pound varies from 72 to 92 lineal yards for ½-in. tape, 50 to 60 yards for ¾-in. tape and 39 to 46 yards for 1-in. tape. The thickness will range from 0.010 to 0.020 in., averaging 0.015 in.

A sheet of cotton cloth weighing from 4 to 6 oz. per square yard is passed through a bath of melted bituminous composition, so that an excess will adhere to the surfaces (from 0.005 to 0.007 in. on *both* sides of the sheet) and after cooling, is torn into strips of the desired width and wound into rolls of predetermined weight. The weight of the product should average 1 lb. per square yard, carrying about 0.33 lb. of cotton fabric and 0.67 lb. of bituminous saturation and coating.

The bituminous composition with which the fabric is treated may consist of one or more of the following products:

Pure native asphalts, residual asphalts, blown petroleum asphalts, wurtzilite asphalt and fatty-acid pitch, a mixture of hard fatty-acid pitch and castor oil,[851] used either singly or in various combinations when of the required consistency, or else if too hard (and the same applies also to asphaltites) fluxed to grade with one or more of the following, viz.: soft native asphalt, residual oil, soft residual asphalt, soft blown petroleum asphalt, soft fatty-acid pitch, animal and vegetable oils and fats, and wool grease.

It should comply with the following tests:

(1) The consistency at 77° F. should be below 7.0 (Test 9*c*).

(2) The susceptibility index (Test 9*b*) should be as low as possible, and preferably under 25.

(3) The ductility at 77° F. should be as high as possible, and preferably over 25 centimeters (Test 10*b*).

(4) The fusing-point by the K. and S. method (Test 15*a*) should be between 80 and 100° F.

(5) The volatile matter at 500° F. in five hours (Test 16) should not exceed 5.0 per cent.

(6) It should appear "tacky" and adhesive at room temperature, and retain this property as long as possible on exposure to air. A strip of tape hung indoors and protected from the direct rays of the sun should show no dimunition in tackiness at the end of two months. This is important, for if the tape once dries out, it becomes valueless.

The following specifications have been formulated: [852]

(*a*) Weight and Dimensions.

Nominal Width	Nominal Thickness (±0.002)	Nominal Weight per Roll	Minimum Net Weight per 100 Rolls*	Minimum Length per Pound
Inches	Inch	Pounds	Pounds	Yards
½	0.015	¼	25	82
¾	0.015	½	50	55
1	0.015	½	50	41
1½	0.015	1 or 1¼	100 or 125	27
2	0.015	1½	150	20

* Exclusive of the core, wrapping and box.

The thickness shall not vary more than ± 0.002-in., and the width ± 1/32-in.

(*b*) Number of Pinholes:

	The Maximum Number of Holes shall not Exceed:	
	In Three Yards	In Any Single Yard
½-inch tape	6	3
¾-inch tape	6	3
1-inch tape	9	4
1½-inch tape	12	5
2-inch tape	15	6

(*c*) Tensile Strength (Test 73*a*): Shall not be less than 40 lb. per inch of width.

(*d*) Adhesion (Test 73*b*): The rate of unwinding shall not be greater than 15 in. in one minute.

(*e*) Adhesion after Heating: The rate of unwinding shall not be greater than 15 in. in one minute.

(*f*) Dielectric Strength (Test 72): The breakdown voltage shall not be less than 1000 volts.

In addition, it is sometimes stipulated that the cotton shall be well saturated, but the compound must not be applied in excess; the surface must be smooth; the edges shall be straight without ravelling; when unwinding from the original roll, there shall be no tendency to leave a thread sticking to the next layer; the weight of

the compound applied to the fabric shall be about 0.65 lb. per sq. yd.; the ash on incineration shall not exceed 15 per cent; the rolls shall be packed in tin foil and enclosed in a metal box to prevent them from drying out.

Variations consist in first impregnating a fabric with a bituminous composition which in turn is surfaced with rubber;[853] or in coating a fabric with an asphalt emulsion and then drying to expel the water.[854]

BITUMINIZED WALL BOARD

Wall board is used in forming ceilings, also partitions between the rooms of dwellings.[855] It is nailed directly to the wooden beams and takes the place of lath and plaster. Wall board is manufactured by assembling two or more layers of pulp board (manufactured from ground wood) or chip board (manufactured from waste papers), so as to form a total thickness approximating ¼ in. The individual layers of pulp or chip board measuring 0.025 to 0.060 in. are generally cemented together with silicate of soda, but in certain cases asphalt is used.[856] The manufacturers contend that asphalt contributes to the moisture-resisting properties of the finished board, but this, however, is a mooted question. When asphalt is used, it is introduced while melted between the several layers of pulp or chip board, following substantially the same procedure as in manufacturing multiple-layered roofings.[857]

Modifications include the following procedures: First impregnating the layers of chip board with asphalt;[858] two layers of board cemented together with an emulsion of asphalt, clay and water to which a fibrous material may have been added;[859] several layers of board cemented together with sodium silicate and waterproofed on the surface with melted paraffin- or montan-wax;[860] two layers of asphalt-saturated felt cemented together with a layer of fibrated asphalt;[861] two layers of asphalt-saturated board cemented together with a layer of gypsum;[862] two layers of paper cemented with an asphalt emulsion to a central layer of gypsum;[863] two layers of pulp board cemented with an asphalt to a central layer of perforated felt;[864] two layers of pulp board cemented together with a mixture of sulfite-cellulose pitch and powdered limestone;[865] two layers of pulp board cemented together with a thick layer of sponge as-

phalt;[866] two layers of granular-surfaced roofing cemented together with hydraulic cement or asphalt in between the granular surfacings;[867] cementing a layer of smooth- or mineral-surfaced roofing with asphalt to one or both sides of wood veneer or wall-board;[868] coating one side of wall-board with asphalt in which are embedded mineral granules;[869] cementing a layer of prepared roofing with asphalt to expanded metal;[870] cementing two layers of chip board with asphalt to wooden laths in the center;[871] etc.

BITUMINOUS INSULATING BOARD

This is used for lining buildings, refrigerating chambers, cold-storage warehouses, refrigerator cars, etc. A number of types have been described, including: a cellular material composed of fibrous material such as straw, grass, wood waste, or the like, treated with melted asphalt or coal-tar pitch without leaving a surplus in the mass, which is then rolled or otherwise compressed in the form of sheets or slabs;[872] mineral wool treated with an asphalt emulsion and molded into sheets;[873] corrugated board cemented with asphalt in between two layers of plain board or prepared roofing;[874] a corrugated product composed of two layers of prepared roofing combined with wire mesh or sheet metal;[875] a corrugated product composed of fibrated asphalt;[876] plain sheets of bituminized fabric;[877] a sheet of fibrous board or felt encased in asphalt;[878] a sheet of cork-board encased in asphalt;[879] two layers of asphalt-saturated paper cemented together with a mixture of asphalt emulsion and vermiculite;[880] alternate layers of hair-felt and asbestos cemented together with asphalt and surfaced with paper;[881] a plurality of felt sheets encased in a bituminized fabric;[882] sheets of perforated bituminized felt alternately cemented together with imperforated sheets;[883] sheets of cloth cemented together under pressure with a mixture of asphaltenes and fillers;[884] a laminated sheet composed of a core of fibrous insulating board cemented with asphalt emulsion or resin to asbestos-cement surfacings;[885] a laminated sheet composed of a core of fibrous insulating board cemented with asphalt to sheet-metal surfacings;[886] etc.

ACOUSTICAL BLOCKS

These are made of saturated-felt wound in convolutions with spacing elements in between.[887]

BITUMINOUS STUCCO-BASE AND PLASTER-BOARD

This product is used as a base for the application of Portland-cement mortar stucco on the outer walls of buildings. The following products have been described for the purpose: a single layer of chip-board surfaced on one side with asphalt, in which are embedded wooden laths, spaced at intervals;[888] wooden laths wired together and coated with asphalt;[889] a layer of chip-board cemented with asphalt to a laminated wall-board base;[890] layers of paper cemented with a bituminous dispersion to a core of gypsum;[891] a layer of chip board fastened with asphalt to another sheet which has been creased and bent to form projections and keyed depressions;[892] a similar structure in combination with wire mesh;[893] mineral-surfaced roll-roofing made in rigid sheets;[894] single-layered or laminated sheets of asphalt-saturated felt cemented together and surfaced with asphalt;[895] asphalt-saturated and coated felt having untreated kraft paper cemented to one surface to which the stucco is applied;[896] sheets of creped paper cemented together with asphalt;[897] a sheet indented to form a plurality of surface projections;[898] celotex surfaced with asphalt in which mineral granules are embedded;[899] a sheet of glass coated with asphalt in which mineral granules are embedded;[900] etc.

CHAPTER XXX

BITUMINOUS LACQUERS, CEMENTS, VARNISHES, ENAMELS AND JAPANS

This chapter will embrace mixtures of bituminous materials with volatile solvents, ranging from pastes to liquids. Bituminous cements are manufactured in paste form, whereas bituminous lacquers, varnishes, enamels and japans are prepared in the liquid state, their respective consistencies being regulated by the amount of volatile solvent incorporated.[1] It should be noted that a mixture of a bituminous base with a volatile solvent, in the absence of drying oils or pigments is classified technically as a "bituminous lacquer," although the trade often erroneously alludes to such compositions under the term "asphalt paint."

BITUMINOUS LACQUERS AND PAINTS

Bituminous lacquers are composed of a bituminous base, a volatile solvent,[2] with or without the addition of vegetable drying oils, resins, mineral fillers and pigments,[3] and are intended to dry or set by the spontaneous evaporation of the solvent. If any vegetable drying oil is present it will contribute little, if at all, to the drying of the lacquer, although it will undoubtedly exert a "toughening" effect on the coating, which will become more pronounced in time. These lacquers are known commercially as "solvent paints." The distinguishing characteristic between a bituminous lacquer and a bituminous varnish is that the former will set to a firm coat upon the evaporation of the solvent, whereas the hardening of the latter is contributed largely by the oxidation of the vegetable drying oil, which is present in the base in substantial proportions.

Nature of the Base. Bituminous lacquers may be divided into four groups, depending upon the composition of the solid ingredients present in the base, viz.:

Group 1. Bases containing a bituminous substance, with or without a mineral filler. The bituminous substances may include: native asphalts,[4] asphaltites including grahamite,[5] asphaltic petro-

leums, residual oil, blown petroleum asphalt, residual asphalt, sludge asphalt,[6] wurtzilite asphalt,[7] wax tailings,[8] mineral waxes, tars, wood-tar pitch,[9] rosin pitch, peat- and lignite-tar pitches, water-gas-tar pitch, oil-gas-tar pitch, coal-tar pitch, fatty-acid pitch,[10] vulcanized fatty-acid pitch,[11] fatty-acid pitch combined with a small percentage of rubber,[12] bone-tar pitch, etc. Products of the group which are rarely used include chlorinated residual asphalt or chlorinated blown asphalt,[13] chlorinated creosote oil,[14] chlorinated montan wax,[15] chlorinated naphthalene,[16] nitrated coal tar,[17] soluble portions extracted from bituminous coal,[18] etc. A mixture of Trinidad asphalt and gilsonite, heated to expel the volatile constituents is claimed to possess special virtues.[19]

Group 2. Bases containing in addition to bituminous substances, a resin (e.g., rosin, rosin ester, damar or sandarac); with or without a mineral filler; with or without a colored pigment.

Hardened rosin[20] adapts itself to being combined with bituminous substances, and a popular form of cheap colored paint consists of a mixture containing gilsonite, hardened rosin (i.e., heated with lime), solvent and colored pigment (usually red iron-oxide). A hard transparent resinous substance is produced from the unsaponifiable constituents derived from coniferous wood tar by heating with formaldehyde.[21] It is claimed that resins of the coumarone-indene group will prevent the settling of free-carbon in solvent paints containing coal-tar pitch or water-gas tar pitch.[22] Other mixtures include the following: fatty-acid pitch, copal, asphalt and solvent;[23] asphalt combined with phenol condensation resins;[24] asphalt combined with rosin heated with sulfuric acid and then neutralized with alkali;[25] asphalt combined with synthetic resin and iron oleate or iron resinate;[26] etc.

The outstanding types of synthetic resins that have recently come into use include the following:[27]

(a) Polyhydric alcohol resins (i.e., glyptals, rezyls and alkyd resins) obtained by reaction of polyhydric alcohols (e.g., glycerol) with polybasic acids (e.g., phthalic anhydride). When combined with vegetable drying oils, the product dries in part by oxidation, and combines the qualities of both lacquers and varnishes. These resins are soluble in coal-tar distillates and the softer forms in petroleum distillates.

(b) Phenol-aldehyde resins (e.g., bakelite) which are generally used in baking varnishes, and may be fluxed with rosin, rosin-ester, and fossil resins, and combined with drying oils (e.g., china-wood oil). The product dries very rapidly by oxidation (4-hour varnishes). Hydroxy-diphenyl and the various higher alkyl-substituted

phenols are soluble in drying oils without first fluxing with rosin, etc., and also dry rapidly.

(c) Vinyl resins (e.g., vinyl acetate) obtained by combination of acetylene and acetic acid in the presence of a catalyst. These consist of a liquid which polymerizes under heat to a resin. They are also produced by polymerization of vinyl chloroacetate and vinyl chloride. Generally used in combination with nitro-cellulose. A similar resin, known as polyvinyl acetal is obtained from normal polyvinyl resin by reaction with an aldehyde, usually formaldehyde or acetaldehyde.

(d) Styrene resins, including polystyrene, have the highest insulation values of any of the synthetic resins, and are soluble in coal-tar distillates, chlorinated hydrocarbons, esters, and cyclohexanone.

(e) Urea resins produced by the action of formaldehyde on urea. Used with nitrocellulose in lacquers. On heating, they rapidly become insoluble and infusible.

Group 3. Bases containing in addition to bituminous substances, animal or vegetable oils or fats, with or without a mineral filler, with or without a colored pigment. The animal or vegetable oils or fats may include: linseed oil,[28] china-wood oil,[29] soya-bean oil,[30] fish oil,[31] cotton-seed oil, corn oil, perilla oil,[32] lard oil, mutton fat, castor oil,[33] cashew-nut-shell oil,[34] oiticica oil heat-treated with sulfur or sulfides,[35] stearic acid, oleic acid, etc. The oils may be used either in the raw state or combined with "dryers," or they may be "bodied" by heating to a high temperature, until they thicken by polymerization. Anti-oxidants (e.g., lead salt of alpha-naphthol) may also be added.[36]

Group 4. Bases containing in addition to bituminous substances, resins in combination with animal or vegetable oils or fats; with or without a mineral filler; with or without a colored pigment. The "resins" may include: common rosin, rosin esters, the damars,[37] (including Borneo damar), sandarac, Kauri copal, Congo copal, pontianak copal and Manila copal.

The following ingredients are rarely included, viz.: wool grease,[38] rosin oil, insoluble metal soaps, beeswax, carnauba wax, Japan wax, paraffin wax, petrolatum, nitrocellulose, cellulose acetate, celluloid, casein and alkaline caseinates;[39] albumin;[40] rubber;[41] chlorinated rubber (soluble in benzene, toluene, tetralin, chlorinated hydrocarbons, and vegetable drying oils, but insoluble in petroleum distillates and alcohols)[42] chlorinated rubber and antimony trioxide;[43] rubber and metallic linoleates (e.g., cobalt or manganese linoleate);[44] synthetic resins including phenol-formaldehyde resins[45] and aldehyde-base resins;[46] synthetic resins mixed with fatty-acid pitch;[47] etc. One method of incorporating rubber consists in masticating crepe rubber with 2½ per cent cobalt linoleate until dis-

persed, and then churning the mixture with an equal weight of petroleum naphtha, which solution may be added to paints and lacquers to improve the brushing and flowing properties.

The anti-corrosive properties of the lacquer may be improved by adding tannic acid;[48] or orthophosphoric acid followed by heat to decompose the acid into pyro- and meta-phosphoric acid (which are less soluble in water);[49] or by adding chromium salts (e.g., $CrCl_3$);[50] or polythioaniline (obtained by heating aniline with sulfur);[51] or heavy metal (e.g., lead or manganese) naphthenates, resinates, sulfonates, linoleates, or their mixtures.[52]

Among the resins: rosin, rosin esters, the damars and sandarac will readily combine with the bituminous constituents, either by dissolving all of them directly in the cold solvent, or by first fluxing them together with heat, and cutting them in the solvent afterwards. Fossil resins (copals) will not amalgamate directly with the bituminous constituents. They must be melted at a high temperature, and first combined with the animal or vegetable oil, whereupon the resulting oleoresinous varnish will flux with the bituminous constituents, either in the cold, or upon melting them together. A description of the process for manufacturing oleoresinous varnishes does not fall within the scope of this treatise.

Since the addition of "vegetable or animal oils or fats" improves the weather-resistance of bituminous substances, they should accordingly be added wherever possible in the rôle of "flux." The harder the bituminous substance in its raw state, and the higher its fusing-point, the larger can be the percentage of the animal or vegetable oils or fats incorporated. Rosin, the damars, sandarac or rubber do not add to the weather-resistance of the bituminous materials, as these are of themselves deficient in this respect. They will either serve to lighten the color of bituminous materials when combined with colored pigments,[53] or in other cases to bring about a more perfect union of the constituents, but in either event they should be confined to indoor use. On the other hand, fossil resins are highly weather-resistant, being equivalent to the asphaltites in this respect. They may accordingly be embodied in bituminous paints intended for outdoor use. Paints containing fossil resins may be made to carry a large percentage of animal or vegetable oils or fats, and will accordingly produce brighter colors with pigments.

The foregoing products may be blended together so the base

will be soft, medium or hard, with a proportionate range in fusing-point. A base which is naturally soft, may be hardened by adding a suitable quantity of mineral filler or pigment. Where the base is allowed to remain soft, the paint will be limited in usefulness, and adapted only to the painting of *porous* surfaces such as masonry or dried-out composition roofings, which will give it an opportunity of soaking into the pores, and thus mask the soft properties of the coating.

Nature of the Fillers and Pigments.[54] All sorts of mineral fillers have been suggested in connection with bituminous paints, for use either alone, or in combination with colored pigments. Their function is to harden the base or to cheapen the paint. The fillers should be powdered very finely (so they will pass a 200-mesh sieve), otherwise they will tend to settle out. The more finely they are powdered and the lower their specific gravity, the longer will they remain in suspension.

The colored pigments include the iron oxides (red oxide of iron),[55] micaceous iron oxide (hematite),[56] red oxide of lead, vermilion, yellow ochre, green oxide of chromium, chrome green (composed of chrome yellow mixed with prussian blue), chrome yellow (lead chromate), zinc yellow (zinc chromate), cadmium yellow, ultramarine blue, cobalt blue, graphite,[57] powdered asphaltite (insoluble in the thinner),[58] powdered anthracite coal,[59] carbonaceous matter extracted from coal-tar pitch,[60] bronze powders [61] including aluminium powder,[62] etc. In the latter connection, it has been found that metallic bronze powders will not leaf in the mixed lacquer indefinitely, due to the gradual solution of the polishing agent from the surface of the powder, consequently, it is desirable to delay mixing the bronze powder until immediately before use. Where the color of the finished lacquer plays an important factor, *intense* pigments should be employed, as these will be required to overcome the dark color of the base. Whites, as for example zinc oxide, titanium oxide, lithopone, white lead, sublimed lead, etc. are rarely employed, but will produce shades of buff and tan. Should free acids be present in the binder, as would be the case if fatty-acid pitch, bone-tar pitch, rosin or rosin pitch were used, the range of pigments is narrowed down to certain metal oxides and lithopone (in the presence of olein or oleic acid) [63] or titanium oxide. Pig-

ments such as chrome green, chrome yellow, zinc oxide, etc., will combine with the free acids and form insoluble compounds, causing the paint to solidify, otherwise termed "gelatinizing" or "livering."

Nature of the Solvents. The solvents ordinarily used for manufacturing bituminous paints may be divided into four groups, viz.:

(1) Petroleum products, including gasoline, naphtha (benzine) and kerosene are most largely used. Among the naphthas there is a series of products boiling within a small temperature range, termed "close-cut distillates," and sold as turpentine substitutes.[64] The gasolines have the lowest flash- and boiling-points and the poorest solvent power, whereas the kerosenes have a higher flash- and boiling-point and a correspondingly greater solvent action; the naphthas falling in between the two. Petroleum ether [65] has also been proposed, but this similarly has poor solvent properties. The solvent properties of petroleum distillates may be improved by adding 4 per cent of butyl alcohol, diacetone alcohol or esterfied glycols.[66]

(2) Coal-tar distillates,[67] including benzol,[68] toluol,[69] solvent naphtha [70] and heavy naphtha.

(3) Solvents derived from the distillation of wood, including turpentine,[71] wood turpentine, rosin spirits, light wood oil, wood creosote oil, pine oil, acetone,[72] acetone oil, etc.

(4) Chemical products, including carbon disulfide,[73] carbon tetrachloride, chloroform, di-chlor methane and tri-chlor ethylene.[74] These evaporate very readily, the carbon disulfide being highly inflammable and the vapors toxic and explosive. The chlorine derivatives similarly evaporate rapidly, but form non-explosive and non-inflammable solvents,[75] and may be used to raise the flash-point of such solvents which are susceptible to ready ignition when used alone.[76] Other chlorine derivatives of a non-inflammable nature include: *s*-trichlorethane or monochlorbenzene;[77] *s*-dichlorethylene, and tetrachlorethylene;[78] pentachlorethane, pentachlorbenzene and hexachlorbenzene;[79] hexachlornaphthalene;[80] mixtures of carbon tetrachloride and monochlorbenzene;[81] etc. The addition of 1 per cent of urea or aniline to solvents consisting of chlorine derivatives, will serve to neutralize any hydrochloric acid that may be liberated on ageing, which might otherwise prove objectionable.[82]

Other solvents include tetralin (tetrahydronaphthalene); decalin (decahydronaphthalene); mixtures of anthracene oil with acetone or alcohol;[83] mixtures of petroleum naphtha with acetone, butyl alcohol or diacetone alcohol;[84] mixtures of solvent naphtha and alcohol;[85] benzyl alcohol (recommended for dissolving a mix-

ture of asphalt, resin and rubber); [86] etc. Grain alcohol, wood alcohol, acetone, and the like cannot be used for dissolving bituminous materials, as they are deficient in solvent properties. It is an interesting fact, however, that the products obtained by treating the unsaturated distillates resulting from the destructive distillation of native asphalts and asphaltites with nitric acid, will readily dissolve in acetone and alcohol.[87]

Hydrogenated petroleum naphthas have a higher solvent power than coal-tar products of a similar boiling-point range. Such products have been produced showing initial boiling points of 200 to 400° F. and final boiling points of 275 to 525° F. The hydrogenation is accompanied by an increase in cyclic bodies and a decided improvement in solvent power in comparison with the original material.

The following table [88] shows the comparative evaporation times of certain commercial solvents, tested by allowing 2 ml. of each material to evaporate under similar conditions from a metal surface 3½ in. square:

	Min.		Min
Carbon disulfide	3½	Crude solvent naphtha	121
Carbon tetrachloride	4½	High-flash naphtha	205
Pure benzol	10	Crude heavy solvent naphtha	290
100 per cent benzol	13½	Heavy naphtha	303
90 per cent benzol	14	Turpentine	142
Straw-colored benzol	18	Wood turpentine	480
50 per cent benzol	23	80° gasoline	4
Pure toluol	29	70° gasoline	8
Commercial toluol	33	66° benzine	16
Straw-colored toluol	36	62° benzine	18
Xylol	89	Petroleum turps substitute (42°)	346
Solvent naphtha	107	Kerosene	475

A more accurate procedure [89] of ascertaining the rate of evaporation of solvents consists in passing a stream of air (dried by bubbling through concentrated H_2SO_4) at a uniform rate of 1 liter per minute over exactly 2 ml. of the solvent in a 200 ml. spherical flask maintained at 77° F. in a water-bath, and observing visually the number of seconds that elapse until the solvent has evaporated completely. The results are expressed proportionately against the time of evaporation of *n*-butyl acetate (C.P.), which is arbitrarily taken as 100. Values of more than 100 indicate faster, and under 100 slower rates of evaporation than that of *n*-butyl acetate. Com-

parable results may be calculated from the empirical formula: $\frac{VM}{11}$; where V = the vapor pressure of the liquid and M its molecular weight; as shown in the following table:

	Observed	Calculated
Acetone	850	975
Ethyl acetate	485	582
Toluol	186	186
Ethyl alcohol (95%)	193	183
n-butyl acetate	100	100
Butyl alcohol	46	32
Amyl acetate	73	85
Cellosolve	37	38

It will be noted that the rates of evaporation are *not* proportional to the boiling points of the respective solvents.

The proportion of solvent used depends upon three factors: (1) the nature of the bituminous base, (2) the "capacity" of the solvent, and (3) the consistency of the product desired.

In general, the higher the susceptibility of the base, the lower will be the viscosity of the resulting paint. Upon running parallel tests, with the same percentage by weight in all cases of the respective solvents, it will be found that residual asphalt, sludge asphalt, wood-tar pitch, water-gas-tar pitch, oil-gas-tar pitch and the coal-tar pitches will form paints of lower viscosity than paints prepared from fluxed asphaltites, blown petroleum asphalt, wurtzilite asphalt and the non-susceptible fatty-acid pitches. With bituminous materials having a high susceptibility index, the fusing-point of the base exerts comparatively little influence on the viscosity of the completed paint. Bases that are hard, and at the same time possess "tough and rubber-like" properties (i.e., a low susceptibility index, considerable elasticity, resilience and tenacity) when used alone, are apt to gelatinize after they have been dissolved in the volatile solvent. In other words, although the paint when first made up may appear liquid, it will after a time become transformed into a jelly-like mass. This is particularly apt to occur with hard and "rubbery" fatty-acid pitches, wurtzilite asphalt and gilsonite. It has been observed, however, that gilsonite may be dissolved without jellying in such solvents as gasoline, by first melting the gilsonite and maintaining it for a sufficient length of time slightly above its melting-point.[90] The presence of free sulfuric acid or sodium sulfate (resulting

from the neutralization of refining acid with alkali) either in the solvent or in the base, has a noticeable effect upon the formation of jellies. It is also claimed in this connection that the loosely combined sulfur present in certain asphalts is converted into sulfuric acid during the blowing process, and that this accounts for the gelatinization of paints containing blown petroleum asphalts as base.[91] It has been suggested that if the asphalt is blown in the presence of lime, this jellying will be retarded. The specific gravity of the base has also a bearing on the paint's consistency. Since it is customary to measure the base and solvent by *weight,* it follows that the higher the specific gravity of the base, the smaller will be its volume for a given weight. Hence a base of high gravity will form a paint of lower viscosity than an equal weight of a low gravity base, but substantially the same in other respects.

The "capacity" of the solvent also has an important bearing upon the viscosity of the paint. The greater the solvent action of the menstruum, the smaller percentage by weight will be required to produce a paint of a given viscosity. In general, aromatic hydrocarbons are superior in solvent action to aliphatic hydrocarbons. Coal-tar products and those derived from wood distillation constitute better solvents than petroleum products. Similarly, distillates from aromatic petroleum are relatively better solvents than those produced from strictly aliphatic petroleum. In all cases the higher the molecular weight of the hydrocarbons, the better will be the solvent action. Derivatives such as carbon disulfide, carbon tetrachloride, chloroform, di-chlor methane, ether, etc., are generally better solvents than the corresponding pure hydrocarbons from which they originated. For the reason previously explained, the specific gravity of the solvent has a bearing on the consistency of the paint, from the standpoint of percentage expressed by weight.

The weight of solvent in commercial bituminous-solvent compositions ranges from 20 to 80 per cent. The smaller percentages are used in heavy-bodied paints intended for coating masonry, for sealing the joints of composition roofing, and for application to porous surfaces. Light-bodied paints containing the larger percentages of solvent are used where it is desired to secure great penetration, rapid drying properties, or where the paint is used for "dipping" purposes.

Mineral waxes alone are difficultly soluble in most solvents. Asphaltites and asphalts dissolve in the following solvents, approximately in the order mentioned, viz.: carbon disulfide, carbon tetrachloride, distillates from coal tar, resinous woods and petroleums. Comparing products of the same fusing-point, we find that blown petroleum asphalts are most soluble, then come native asphalts, sludge asphalt, wurtzilite asphalt, and lastly the residual asphalts.

Tar pitches produced from the destructive distillation of bones, wood, peat, lignite and coal are more difficultly soluble than asphaltic materials and pitches not derived from tars (e.g., rosin pitch and fatty-acid pitch). Tar pitches dissolve most readily in the following solvents, approximately in the order mentioned, viz., carbon disulfide, coal-tar distillates and distillates from resinous woods. Of the pitches: rosin pitch, fatty-acid pitch,[92] bone-tar pitch, peat- and lignite-tar pitches are relatively the most soluble, and oil-gas-tar pitch, water-gas-tar pitch, wood-tar pitch and coal-tar pitch are least soluble, ranging approximately in the sequence stated. The solubility of bituminous substances may be increased by fluxing with such materials as rosin, animal and vegetable oils or fats, wax tailings, fatty-acid pitch, and in some cases residual oil derived from aromatic petroleums.

Although coal-tar pitch is largely insoluble in petroleum naphtha, the addition of 1 to 3 parts of tar acids will cause 80 parts of the pitch to form a colloidal solution with 20 parts of petroleum naphtha, showing no tendency to separate.[93] The separation of free carbon may likewise be hindered by dissolving the melted coal-tar pitch in solvent naphtha and heating the solution betwen 100 and 200° C.;[94] or by adding coumarone resin to the solution;[95] or by thoroughly stirring the solution and heating under pressure.[96] A product may on the other hand be obtained from coal-tar pitch which will withstand the action of petroleum products, by dissolving the pitch in benzol, filtering off the free carbon, distilling the benzol, and finally extracting the residue with petroleum naphtha until all the soluble matter has been extracted. The final residue is insoluble in petroleum products, but may be readily dissolved in benzol or coal-tar naphtha.[97] Wood-tar pitch mixed with fillers is also recommended to withstand the action of petroleum products.[98] A mixture

of gasoline, or turpentine with alcohol has been suggested for dissolving coal-tar pitch.[99]

In the manufacture of emulsion compositions, the foregoing solvents are replaced by water, in which the bituminous binder is dispersed by any of the emulsifying or dispersing agents described in Chapter XXV.

Methods of Manufacture. When all the ingredients of the base fuse at comparatively low temperatures, the simplest method consists in stirring them with the solvent in a closed steam-jacketed tank provided with an agitator. The temperature should be raised close to the boiling-point of the solvent, and the stirring continued until the solution is complete. When some of the materials melt at high temperatures it is advisable to flux them with the remaining ingredients of the base by direct fire, either in a small tank mounted

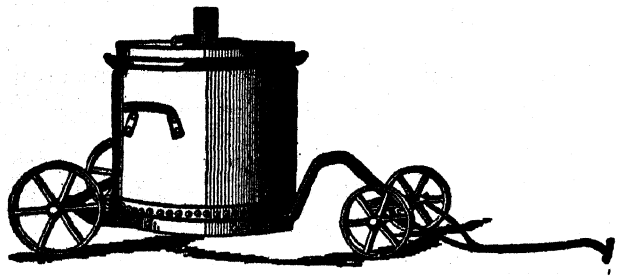
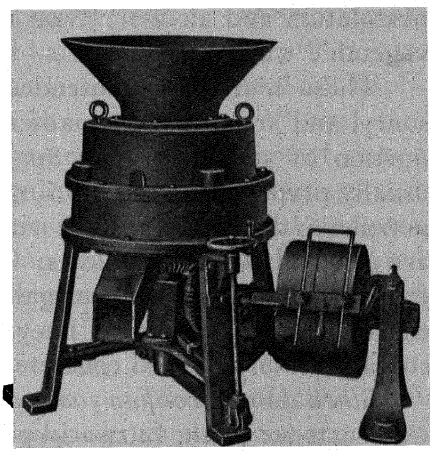


FIG. 191.—Varnish Kettle.

on wheels, known as a "varnish kettle," heated by coke burning in a pit, on grate bars sunken 8 to 12 in. below the floor level (Fig. 191), or in a large stationary hemi-cylindrical melting tank. When the melting is effected in a varnish kettle, after the ingredients have been fluxed together, it is withdrawn a safe distance from the fire, cooled to slightly under the boiling-point of the solvent, which is then gradually introduced, small quantities at a time, stirring continuously with a paddle. When the base has been melted in a stationary hemi-cylindrical kettle, it is run by gravity or pumped into a closed cylindrical tank equipped with a stirring device, and after cooling sufficiently, the solvent is run in slowly, with continuous stirring until the solution is complete. The second method is used for preparing large quantities of lacquer in one charge. Where the lacquer is to be made from a high fusing-point asphalt or asphaltite

the solution may be effected by stirring or otherwise agitating the base—preferably ground to a fine mesh—with the solvent in the cold for a period of time.[100]

Sometimes the bituminous lacquer is mixed cold in various proportions with oleoresinous varnishes manufactured in the usual manner, either to increase its weather-resistance or for purpose of lightening its color, when colored pigments are to be incorporated. Pigments and fillers may be mixed with the paint by simply stirring them together in a tank provided with an agitator, or by grinding the mineral ingredients and sufficient paint to form a paste in an enclosed buhr-stone mill, as illustrated in Fig. 192, and then thinning the paste to brushing consistency with an additional quantity of bituminous paint, or in certain cases with a volatile solvent. Fillers or pigments ground in accordance with the latter procedure are less apt to settle on standing, than if simply stirred into the paint. It should be noted, however, that even under the best conditions, fillers and pigments are more apt to settle from bituminous solvent compositions than from oil paints. This is probably due to the better buoying and lubricating properties of the oil on the mineral particles.



Courtesy of Kent Machine Works.

FIG. 192.—Paint-grinding Mill.

The use of pebble mills results in a finer grinding of the pigments and fillers, and recently colloidal mills have been finding favor, since they seem to give even better results than any of the older types of grinding apparatus.

Masonry Coatings. These are used for painting brick, stone, or concrete surfaces,[101] usually above ground, to prevent the inroads of dampness or moisture. Masonry coatings often termed "damp-proofing paints," are divided into two classes, viz., clear and black.

Clear damp-proofing coatings are used for painting the outside of exposed walls, and are generally made up from a base composed largely of paraffin wax, beeswax, candelilla wax, carnauba wax or

Japan wax, or mixtures of these waxes in combination with resinous, oily or fatty constituents.[102] Paraffin wax alone is only slightly soluble in most solvents, and has the disadvantage of crystallizing out at low temperatures. The addition of resins, vegetable or animal oils and fats will prevent this to a certain extent, and also maintain the necessary light color. As an example, it may be cited that paraffin wax fluxed with china-wood oil will dissolve in about twice its weight of petroleum naphtha;[103] also a mixture of paraffin wax, petrolatum and alcohol;[104] or an aqueous dispersion of mineral or vegetable wax.[105]

These coatings are intended to penetrate the pores of the masonry, and are therefore made thin in body, containing a large proportion of solvent (60 to 80 per cent by weight). The base is usually prepared of a low fusing-point to augment its solubility, and petroleum distillates, with or without the addition of wood distillates are generally used as solvents. Clear damp-proofing coatings spread from 250 to 500 sq. ft. per gallon, depending upon the porosity of the underlying surface. They are also recommended for use in coating freshly poured concrete to promote curing.[106]

Black damp-proofing coatings are made from asphaltic mixtures, water-gas-tar pitch, fatty-acid pitch and the various coal-tar pitches, used either alone or in combination with animal or vegetable fats or oils. The base is prepared of a fairly low fusing-point (140 to 160° F. by the K. and S. method) and "cut" to a heavy body. Asphaltic bases are dissolved in 40 to 60 per cent of petroleum distillate figured on the total weight; and bases derived from tars dissolved in 30 to 45 per cent of coal-tar distillate.[107] This class of coatings is used for coating the *inside* of exposed masonry walls, and may be plastered upon *directly,* thus dispensing with the furring and lathing, and are hence often termed "plaster bond."[108] The best practice consists in applying two coats of the damp-proofing coating to the inside of the wall by means of a brush or a spray-gun,[109] allowing each to dry about twenty-four hours. The plaster may then be trowelled directly on the paint, with which it forms a good bond. Black damp-proofing coatings containing saponifiable materials such as fatty-acid pitch, vegetable or animal oils and fats, or resins, bond better with the plaster than those made exclusively

of asphaltic or coal-tar mixtures, in which latter case the bond tends to weaken when the coating ages. The coating should spread between 50 and 60 sq. ft. per gal. for the first coat, or 70 and 80 sq. ft. for the second, depending upon the character of the surface to which it is applied.

Other mixtures recommended for this purpose include: asphalt or coal-tar pitch with a saponified vegetable oil (e.g., cottonseed oil), which is heated to 400° F. and blown until thoroughly oxidized, or as an alternative vulcanized with sulfur, and in either case dissolved in petroleum naphtha;[110] cut-back coal-tar pitch;[111] a mixture of water-gas tar and paraffin wax dissolved in coal-tar naphtha;[112] asphalt, ozokerite, paraffin wax and solvent;[113] coal tar, montan wax and solvent;[114] coal tar treated with an excess of slaked lime and mixed with sodium silicate;[115] sulfurated water-gas tar mixed with a metallic bronze powder;[116] asphalt or coal-tar pitch dissolved in a solvent and after being applied, having granulated cork or mineral particles applied to the surface;[117] first applying a wash of cement grout to the masonry surface, followed by the bituminous coating;[118] etc. Bonding to damp surfaces may be improved by adding ethyl alcohol to the petroleum solvent.[119] A modified procedure consists in applying an aqueous dispersion of asphalt by means of an air-gun, the first coat being mixed with Portland cement to stop suction of the masonry, subsequent coats being applied without such admixture.[120]

Stone Backing. Bituminous paints similar to the black dampproofing coatings described in the previous section are used for coating all the surfaces except the exposed face of cut stone used in the construction of buildings, to prevent staining or efflorescence as well as other forms of discoloration from the ingredients of the mortar. Other mixtures recommended for this purpose include: paraffin wax, beeswax, linseed oil and solvent;[121] coal tar, rosin and powdered shale;[122] petroleum sediment (known as "B.S."), solvent and pigment.[123]

Asphalt Primer for Use in Built-up Roofing and Waterproofing. This is used for priming concrete surfaces prior to the application of the first mopping coat of melted asphalt in laying built-up roofings or membrane waterproofings involving the use of asphalt-

saturated felts, so as to promote the bonding of the asphalt with the concrete roof deck or masonry surface. The following specification [124] has been proposed for such primers:

Asphalt base	Min. 35 per cent by weight
Softening-point (R. & B. method)	131 to 170° F.
Penetration at 77° F., 100 g., 5 sec.	15 to 50
Solvent:	
Distillate under 248° F.	Max. 20 per cent by weight
End-point	Max. 500° F.

Paints for Resurfacing Prepared Roofings. These fall into two groups, namely those intended to repaint prepared roofings composed of asphaltic and coal-tar products respectively. For the former, asphaltic compositions are used, and for the latter, compositions prepared from pitches, and generally coal-tar pitch.[125] For the cheapest coating, straight-distilled coal tar or crude asphalt-bearing petroleum is used. These form coatings of a nondescript character, due to their variable composition, and they also evaporate and harden slowly. The characteristics of coal tar or shale tar are claimed to be improved by heating it with hydrated lime,[126] or mixing it with aluminium resinate,[127] or sawdust.[128] A mixture of wood tar and sulfite liquor has also been recommended; [129] likewise a mixture of lignite tar, wood tar, rosin, rosin oil and sulfur; [130] coal tar and montan wax; [131] coal tar, creosote oil, asphalt and linseed oil; [132] coal tar, rubber and solvent; [133] coal tar and carbonized cotton fibers; [134] coal tar, asbestos, mica and graphite with a solvent; [135] coal tar mixed with casein and mineral fillers and a volatile solvent; [136] fatty-acid pitch and rubber dissolved in a solvent; [137] etc.

Higher grade coatings of this class are prepared from substantially the same compositions used for the coatings of prepared roofings, but of a slightly softer consistency and lower fusing-point (140 to 160, K. and S. method). These are dissolved in approximately an equal weight of solvent.

When used as lap-cement, a smaller proportion of solvent is incorporated, and the paint is accordingly made heavier in body. The addition of 1 to 3 per cent by weight of trichlorethylene is claimed materially to increase the adhesive and binding properties of the paint, when used to cement the laps of prepared roofings.

In all cases, it is important that the base should be *plastic,* and have the same physical characteristics as the bituminous roofing to

which it is applied. Coatings containing a preponderance of "vegetable drying oils" such as linseed, china-wood, etc., fail to answer satisfactorily on bituminized roofings, for the following reasons:

(1) Vegetable drying oils oxidize to an *elastic* substance known as "linoxyn," entirely devoid of plasticity, with the result that such coatings soon crack, check ("alligator"), and in certain cases peel off the roofing, when exposed to the weather.

(2) Because the dark-colored bituminous constituents have the property of diffusing or drawing through a coating of dried or oxidized vegetable oils, upon being subjected to the sun's heat, thereby causing the paint to assume a badly discolored or mottled appearance.

Bituminous materials interfere with the oxidation of vegetable drying oils, and if an excess of the former is present in the base, the entire composition will assume a "plastic" consistency, and the drying of the oil will be retarded. On the other hand, if the vegetable drying oil is in excess, it will oxidize in spite of the bituminous matters. The following table shows the relation between the hardness at 77° F., and the percentage of commercial boiled linseed oil in admixture with gilsonite.

TABLE LXXVII

Gilsonite, Per Cent	Boiled Linseed Oil, Per Cent	Consistency at 77° F. (Test 9c)	Penetration at 77° F. (Test 9b)	Remarks
100	0	100 0	0	Unsuitable for coating prepared roofings.
95	5	98.0	½	
90	10	85.5	1	
85	15	70.0	2	Probable range permissible.
80	20	50.0	5	Most desirable range
75	25	20.5	25	
50	50	3 1	220	
25	75	0.0	Too soft	Unsuitable for coating prepared roofings.
15	85	0.0	Too soft	
0	100	0.0	Too soft	

Many unsuccessful attempts have been made to adapt linseed-oil-pigment paints for coating prepared roofings, as for example, by first sizing the saturated sheet with starch paste or the like;[138] or using a mixture of pigment or solvent, in the expectation that the latter will soften the bituminous coating and thereby act as a binder;[139] applying a priming coat of rosin, Portland cement and solvent;[140] surfacing the roofing with fine sand;[141] priming the roof-

ing with aqueous sulfite cellulose;[142] priming with a mixture of rosin, alcohol and lithopone;[143] with coumarone resin dissolved in benzol and petroleum naphtha;[144] with a mixture of coal tar, rosin, clay and powdered mica;[145] with a mixture of coal tar, rosin, vegetable drying oils, barium sulfide, zinc sulfide and coal-tar naphtha;[146] also by treating the underlying asphalt coating with metallic oxides and linseed oil, heated together at high temperatures.[147]

To coat prepared roofings in colors successfully, a weatherproof *plastic* bituminous binder must be selected, having a light brown color in a thin layer (1–2 mils thick) when viewed by transmitted light on glass; otherwise yielding a brown streak on porcelain (Test 6). Gilsonite and certain forms of fatty-acid pitch (Group 1); mixtures of these with certain resins (Group 2); mixtures of these with a small proportion of vegetable oils or fats (Group 3); or mixtures containing both resins and vegetable drying oils (Group 4), will answer satisfactorily for this purpose, since they may be mixed with pigments without obliterating their color, as would be the case if dark-colored bituminous mixtures yielding a black streak were used.[148] The base is dissolved in the volatile solvent and finally the colored pigment or mixture of pigments ground in. The ratio of pigment will vary from half to the same weight as the base, depending upon the intensity of the pigment, the opacity of the base, and the color of the paint desired. The percentage of volatile solvent must be increased slightly when pigments are used, due to that fact that these contribute to the body of the paint. A typical mixture will contain 30 per cent base, 20 per cent pigment, and 50 per cent volatile solvent.

Other compositions recommended for coating composition roofings so as to produce colored effects include: spirit varnishes mixed with colored pigments;[149] a mixture of cellulose nitrate and castor oil dissolved in acetone and ethyl alcohol and containing a colored pigment;[150] a similar mixture containing cellulose nitrate and aluminium resinate;[151] cellulose-ester lacquers containing metallic bronze powders;[152] a mixture of fatty-acid pitch, olein, pigment and solvent;[153] a mixture of gilsonite, Trinidad asphalt, solvent and pigment;[154] a mixture of coal-tar pitch, wood tar, linseed oil and pigment;[155] coal tar and red iron-oxide;[156] coal tar with colored mineral pigments;[157] rosin, paraffin oil and red iron-oxide;[158] phe-

nol-aldehyde resin, vegetable drying oils, pigment and thinner;[159] potassium silicate, caustic potash, chalk and mineral pigments,[160] etc. Emulsion paints containing a bituminous base have also been used successfully for this purpose (see Emulsion Paints).

Asphalt Fibrous-roof-coating. This consists of a heavy-bodied mixture of asphalt, short-fibered asbestos and solvent, sometimes containing a filler. It is intended for recoating smooth-surfaced roll-roofings and metal roofs, and shall comply with the following specifications:[161]

Condition in Container: The material shall not liver in the container and after slight stirring shall be a smooth homogeneous mixture.

Consistency: It shall be of a heavy brushing consistency to insure a film of appreciable thickness and yet not so stiff as to prevent easy application with a 3-knot roof brush at the rate of approximately 1 gallon per 50 sq. ft. over asphalt roofing.

Non-volatile Matter: The roof coating shall yield not less than 60 per cent non-volatile matter when 10 grams are heated in an oven at 221° F. for 24 hours.

Mineral Filler: The material shall contain not less than 5 per cent and not more than 25 per cent of a suitable mineral filler. The residue obtained from the ignition of the mineral filler shall be not less than 85 per cent.

Behavior at 140° F.: Test pieces prepared by brushing the coating on smooth-surfaced asphalt prepared roofing and metal at the rate of 1 gallon to 50 sq. ft., exposed for 60 minutes in a well-ventilated room and then in an oven at 140° F. for 5 hours shall show no blistering, and not more than ¼-inch sagging or slipping, and no appreciable absorption of the coating by the prepared roofing.

Behavior at 32° F.: Metal test pieces from the preceding when bent quickly over a 1-inch mandrel immediately after exposure to a temperature of 32° F. for 1 hour shall show no cracking of the coating and no separation of metal and coating.

Acid-resisting Coatings.[162] Bituminous compositions have been used with outstanding success for the protection of concrete floors, vats, tanks, and other concrete or wooden surfaces against the destructive action of mineral acids, acid salts, alkalies, and other corrosive chemicals. In fact, they are more resistant to these agents than any other type of paint at present known. Two kinds are marketed, namely those made with an asphalt and those with a coal-

tar pitch base, thinned to the desired brushing consistency with suitable volatile solvents. For very dilute mineral acids, coatings made from either base are suitable, but asphalt lacquers appear to be somewhat more resistant than the coal-tar lacquers against acids stronger than 10 per cent. Asbestos fiber is sometimes added to increase the thickness of coating obtained.

Bituminous lacquers will not resist abrasion and their effectiveness depends upon the thickness and imperviousness of the dried coating. Portland cement is very susceptible to acid of any strength, and consequently any pin holes due to faulty application or any abrasion in the surface will be sufficient to destroy the protection afforded by the coating. Once the acid penetrates the coating it will rapidly soak into and disintegrate the concrete beneath, and cause the coating to form blisters which will ultimately flake off.

The most satisfactory method of applying bituminous-solvent compositions is to use a thin priming coat to fill up the surface pores and thus obtain a good bond with the concrete surface, and when this is thoroughly dry, to apply carefully at least two coats of a thicker material of the same character, or containing asbestos fiber, allowing sufficient time for the first coat to dry before the application of the second. The primer can be made by thinning the thicker material with a suitable volatile solvent. A petroleum distillate such as mineral spirits or turpentine substitute is generally used for asphaltic lacquers, and coal-tar distillates as solvent naphtha for the coal-tar lacquers. It is important that the concrete surface be thoroughly dry and fairly dust free. Special care should also be taken to touch up and recoat all dull, brown, porous, and uncoated spots. At least one week should elapse after the application of the last coat before exposure to the acid.

A specification upon which satisfactory material has been obtained is as follows:[163]

General: Shall not settle, thicken or liver.
Color: Jet black.
Free Carbon: Max. 0.5 per cent insoluble in benzene.
Mineral Matter: Max. 1 per cent.
Manganese: Max. 0.10 per cent.
Lead: Max. 0.02 per cent.
Benzene: None.
Resistance to Heat: Shall not soften after 1 hour at 212° F.

Toughness: Dried film on metal shall not chip or scale upon bending over a 5-mm. rod.

Resistance to Water: Shall withstand cold water 18 hours.

Resistance to Acids: Shall withstand for 6 hours: picric acid saturated solution; sulfuric acid 1.3 sp. gr.; nitric acid 1.22 sp. gr.; hydrochloric acid 1.09 sp. gr.

	Type I	Type II	Type III
Non-volatile matter	37% ± 3%	52% ± 3%	82.5% ± 3%
Set to touch	15 min.	2 hours	8 hours
Dry hard	45 mins.	8 hours	24 hours
Consistency	Brushing or spraying	Brushing	Pasty
Penetration at 77° F	.....	.....	200 ± 50

The acid-resisting properties of the composition are stated to be improved by first pulverizing the bituminous base at low temperatures and then subjecting the resulting powder to the action of mineral acids for a lengthy period of time, before it is dissolved in the solvent;[164] or by first nitrating the bituminous base (e.g., nitrated coal tar).[165] The alkali-resistance may similarly be improved by treating the substance (e.g., coal tar) with caustic alkali.[166]

Bituminous Coatings for Metal or Wood. All sorts of bituminous mixtures have been recommended for painting metal and wooden surfaces.[167] A well-known formula consists of a mixture of asphaltite with fatty-acid pitch dissolved in a volatile solvent, known to the trade as "Brunswick black."

Bituminous coatings give better results out-of-doors on metal than on wooden surfaces, although most manufacturers recommend them indiscriminately for both types of construction. However, it should be noted that any resinous constituents in the wood will act deleteriously on the coating after a time, due to their solvent action on the bituminous constituents present. Bituminous varnishes and enamels may safely be recommended under these conditions, since the excess of drying oil on oxidation becomes immune to the resinous constituents. If manufactured according to correct principles, bituminous lacquers will give fair service on exposed metal work, including metal roofs, corrugated iron, farm implements, etc., and although inferior in weather-resistance to bituminous varnishes and enamels, they are at the same time considerably less expensive.

Bituminous lacquers have been used successfully for submerged metal work,[168] although better results will be obtained if the metal is first primed with a red-lead linseed oil primer.[169] It is recommended that metal tanks be protected by a coating of bituminous lacquer in which sheets of aluminium foil are embedded.[170]

Bituminous coatings will not give satisfaction when exposed to vapors or the direct contact of solvents for bituminous materials, such as petroleum products, illuminating gas, the so-called "gas-drip" condensing out of gas mains, turpentine, exudations of resinous woods, or the like. They may be applied on top of dried coatings of linseed oil paint or varnish, but should not be recoated therewith, as bituminous materials will form an unstable foundation, and for the reasons previously cited will act injuriously on the superimposed coating of dried linseed oil (linoxyn). It has been proposed that bituminous lacquers (e.g., tar paints) which dry to a soft, tacky coating may be hardened by spraying with S_2Cl_2.[171]

Certain bituminous compositions, prepared without the use of mineral fillers or pigments, are used for electrical insulating purposes where rapid "air-drying" is required. They are usually marketed under the erroneous term "air-drying varnish," but are in reality air-drying lacquers. The author has examined specimens capable of withstanding 1000 volts per mil in thickness. When coal-tar pitches are used, the insulating properties may be improved by filtering off the "free carbon" after dissolving in the solvent.[172] Alkaline salts of fatty-acid pitch dissolved in a volatile solvent are also claimed to have excellent insulating properties.[173]

For manufacturing inexpensive machinery and farm implements, bituminous lacquers may be used for dipping purposes, upon thinning with additional solvent. An asphalt-wax composition has been proposed for coating the interior of beer cans.[174]

Anti-fouling Paints.[175] Pigmented bituminous lacquers have been used to protect the bottom of metal ships against the corrosive influences of sea water and the growth of barnacles and sea-weed. These belong to the class of "marine anti-corrosive paints" and are composed of a mixture carrying a pigment of toxic properties toward marine growths. The following compositions have been used: gilsonite, fatty-acid pitch and bodied china-wood oil with a

toxic pigment, dissolved in solvent naphtha or "tar oil";[176] asphalt, paraffin oil, wood tar and copper arsenite;[177] coal-tar pitch dissolved in chlorbenzene and tetrachlor-ethylene with the addition of copper or mercury soaps and red iron-oxide;[178] coal tar with arsenic and borax;[179] coal-tar pitch, wood tar, sulfur, concentrated alkali, creosote oil and fillers;[180] etc. The most efficient toxic ingredients consist of either mercury or copper oleate, linoleate, tungstate, or resinate. These remain permanently in solution in the usual paint solvents. Mercury salts are the more efficient, but at the same time are more expensive than the copper salts.

Bituminous Emulsion Paints. These may be prepared by any of the methods described in Chapter XXV. The dispersing agents generally employed are colloidal clay, soaps, and alkaline caseinates. Emulsion paints may be made with or without the addition of colored pigments or dyes,[181] including the use of bentonite dyed with an organic dyestuff,[182] and in certain cases with metallic bronze powders.[183] To prevent freezing in cold weather, methyl alcohol, acetone, glycerin, or glycol may be added. Present-day practice consists in first mixing the components in an agitator and then running them through a colloid mill as previously described. Bituminous emulsion paints are used for coating masonry, prepared roofings, metal and wood. Asphalt-fiber coatings and bituminous cements may also be prepared in this manner, but with these the asbestos fibers are introduced last by a process involving simple stirring. Emulsion paints have been patented for use as a stucco base coat,[184] and emulsion cements for sticking together the layers forming built-up roofs, which may thereby be constructed in the cold.[185] When used for protecting metal, it is recommended that a rust-inhibitor, such as red-lead, litharge, or blue-lead concentrate be used as pigment;[186] or else metallic chromates, borates or phosphates.[187] It has also been suggested that metal surfaces (e.g., steel pipes) be protected by applying a layer of asphalt emulsion, followed by an application of a slury composed of Portland cement and water, which in turn may have a proportion of asphalt emulsion admixed.[188] The use of a cut-back asphalt applied over the bituminous emulsion has also been proposed.[189] Emulsion paints containing toxic ingredients (e.g., insoluble mercury soap dissolved in the asphalt) are recommended for anti-fouling purposes.[190]

It is claimed that the resistance of emulsion paints to the weather may be increased by moistening the surface, after evaporation of the water, with a solvent which will "cut" the bituminous constituents.[191] The incorporation of a solvent (e.g., petroleum distillate, benzol, carbon tetrachloride, acetone, turpentine, methyl-ethyl ketone, methyl acetate, amyl acetate, etc.) with the emulsion, in conjunction with the water, has also been proposed.[192]

Flooring Compositions for Cold Application.[193] Asphalt flooring compositions for cold application are of trowelling consistency and consist essentially of a relatively hard asphalt, asbestos fiber, and finely powdered siliceous mineral filler, thinned with a volatile solvent. When the solvent evaporates, a very thin coherent coat of the intimately mixed asphalt and mineral matter is left behind.

Asphalt flooring compositions are used chiefly where a thin surfacing for concrete floors is desired, or over asphalt-mastic floors [194] as in laboratories which do not have to withstand any traffic heavier than walking. The black composition is most resistant to acids, but where appearance is of importance, red, brown, and olive green are sometimes used. In that case only the top two or three coats of the composition shall contain pigment.

In general, four different compositions are used in applying these coatings: (1) primer, (2) body coat, (3) neat or finishing coat, and (4) floor dressing.

The primer consists of about three parts of asphalt dissolved in seven parts of a volatile solvent. It is of fairly thin consistency and dries to a tacky state in from one-half to three hours, in which condition it is suitable for the application of the succeeding coats. The asphalt used in the primer is similar to that used in the body coat and finishing coat and has a melting-point (Ring and Ball Method) of 88 to 116° C. (190 to 240° F.) and a penetration at 25° C. (77° F.) of about 10.

The body coat is always black in color, is of heavy trowelling consistency, and dries hard in from twelve to forty-eight hours. It consists of asphalt, asbestos fiber, small amounts of fine siliceous mineral matter, and a suitable volatile solvent.

For colors other than black a composition called the first colored top coat is used directly on top of the body coat and before the application of the neat or finishing coat. It dries hard in about

the same time as the body coat and consists of asphalt, asbestos fiber, pigment, and volatile solvent.

The neat or finishing coat may be black, red, olive green or brown. It usually contains no asbestos. The black composition consists of asphalt, fine siliceous material, and solvent. The colored compositions contain pigments instead of part or all of the fine siliceous material. Pulverized rock-asphalt and pigment has also been recommended for this purpose.[195]

The floor dressing is a very thin liquid of a varnish-like character and is applied like a varnish or floor dressing over the completed floor.

From the analysis of compositions of the above types, the following general formulas have been obtained:

TABLE LXXVIII

Constituents	Primer	Body Coat	First Top Coat (Red)	Neat or Finishing Material Red
	Per Cent	Per Cent	Per Cent	Per Cent
Mineral spirits or other volatile solvent.	65–70	33–42	30–36	25–32
Asphalt	30–35	12–16	8–20	10–11
Asbestos and fine siliceous material	.....	42–55	20–22	0–5
Pigment	.....	.....	30–34	57–60

Floor Dressing	Per Cent
Mineral spirits	70–85
Non-volatile (oil or resin)	15–30

For the application of a floor of this type over concrete the following procedure is generally used: The clean, dry, dust-free concrete is given one coat of the primer with a brush and when this has dried to the tacky state, which requires usually from one-half to three hours, a thin layer, about 0.8 mm. (1/32 in.) thick, of the body-coat composition is trowelled on and smoothed out. When this has dried for twenty-four to forty-eight hours, it is rolled with a light wood or iron roller, and successive 0.8 mm. (1/32 in.) coats of the body composition are applied until the required thickness is obtained. The floor is then finished with one or two 0.8 mm. (1/32 in.) coats of the neat or finishing material. In the case of

colored floors, a coat of the first colored top composition is substituted for one coat of the body coat composition.[196] When the completed floor has dried for about forty-eight hours it is given a final rolling and then a thin coat of the floor dressing, which dries to a gloss and prevents dusting, or it can be given a coating of a good floor wax applied in the ordinary way.

It has also been proposed to manufacture flooring of this character in sheet form by applying successive layers of the mastic to a web of asbestos felt, and after allowing same to dry, calendering the sheet to a smooth and uniform structure.[197] Other mixtures include: a pigmented asphalt emulsion containing mineral fillers (e.g., Portland cement) and pigments,[198] likewise a similar composition containing in addition rubber.[199]

BITUMINOUS CEMENTS

These are of plastic, trowelling consistency and adapted for repairing metal or composition roofing, damp-proofing the inside of masonry walls above ground, and to a limited extent for waterproofing the outside of foundation walls below ground. They are sometimes known by the name "asphalt putty." The cement may be composed of two or more of the following constituents, viz.:

(1) A base containing one or more bituminous materials including asphalts and coal-tar pitch, with or without the addition of vegetable oils, resins, etc. The ingredients should preferably be blended together in such proportions that the mixture will show a fusing-point (K. and S. method, Test 15*a*) between 135 and 175° F., a consistometer hardness (Test 9*b*) of 5 to 25 at 77° F., a susceptibility index (Test 9*d*) below 25, and substantially complete solubility in the solvent used.

(2) Mineral fillers are sometimes added, comprising any of the products, such as clay,[200] cement,[201] etc., used for filling the coatings of prepared roofings. Occasionally colored mineral pigments are used to impart to the cement, a color other than the inherently dark color of the bituminous materials. Even when employed in a moderately large proportion, and of a strong intensity, they will result in comparatively dark hues.

(3) Fibrous matter is often added to bind the base together, and form a tougher mass when set. The fibers may either be of mineral, vegetable or animal origin, including asbestos,[202] slag wool, cotton flock, shoddy, rag fibers, sawdust,[203] cotton or wool fibers,[204]

blue or yellow asbestos fibers (known as "amosite," which is mined in Griqualand, South Africa),[205] etc. These are added in percentages not to exceed 25 per cent of the total. The waste product obtained in the manufacture of bituminized roofing or shingles may also be used for this purpose.[206]

(4) A volatile solvent in which the base will dissolve. This may consist of petroleum products (e.g., gasoline, naphtha, or kerosene),[207] distillates from rosin or wood (e.g., turpentine, wood turpentine, pine oil, wood-creosote oil, etc.), tar distillates (e.g., benzol, coal-tar naphtha, solvent naphtha, creosote oil, etc.), used singly or in various combinations. The percentage of solvent will range from 20 to 30 per cent in weight, depending upon the nature of the base and the desired consistency of the cement.

The addition of a water-insoluble soap, such as calcium linoleate or stearate or resinate, zinc oleate, or aluminium palmitate [208] emulsified with water, is claimed to improve the easy spreading properties of the composition,[209] and at the same time facilitate the adhesion to damp surfaces.[210] The addition of pine oil or crude pine tar, or rosin,[211] or animal or vegetable fats [212] similarly improves the spreading qualities and makes the cement less susceptible to temperature changes.

The cement is ordinarily applied with a trowel in a layer from 1/16 to 1/8 in. thick, covering 25 to 12 1/2 sq. ft. respectively per gallon on a fairly smooth surface. The volatile solvent should evaporate within twenty-four hours, leaving the base and any mineral filler or fibrous constituents in the form of a weather-proof coating capable of withstanding the highest sun temperatures without softening or running, and sufficiently tough to withstand walking, attrition of the elements, or any expansive or contractive strains to which it may be subjected.

The following specification has been proposed: [213]

General: Shall be composed of a suitable inorganic filler, a suitable solvent, and a non-volatile bituminous binder, which shall either be mainly asphaltic, or mainly coal-tar.

Condition in Container: The plastic cement shall be a smooth uniform mixture, not thickened or livered, and showing no separation which cannot be easily overcome by stirring.

Non-volatile Matter: The plastic cement shall yield not less than 70 per cent of non-volatile matter when 10 grams are heated in an oven to 221–230° F. for 24 hours.

Inorganic Filler: The plastic cement shall yield on ignition not less than 15 per cent, nor more than 45 per cent of ash.

Workability: The plastic cement shall spread readily without drawing or pulling when applied with a trowel to prepared roofing, asphalt-saturated felt, and to metal surfaces.

Behavior at 140° F.: Test pieces prepared as in the preceding on smooth-surfaced prepared roofing, asphalt-saturated flashing felt and steel, and exposed for 60 minutes in a well-ventilated room and then in an oven at 140° F. for 5 hours shall show no blistering and not more than ¼-in. sagging or slipping, and no appreciable absorption of the cement by the prepared roofing or saturated felt.

Behavior at 32° F.: Metal test pieces from the preceding, when bent quickly over a mandrel 1-in. in diameter, immediately after exposure to a temperature of 32° F. for 1 hour, shall show no cracking of the cement, and no separation of metal and cement.

A dispersed product has been proposed consisting of an asphalt emulsion containing asbestos fibers, or organic fibers,[214] to which may be added an alkali hydroxide or tri-alkali phosphate to prevent the premature breaking of the dispersion;[215] or else a suspension of clay containing acetic acid, propionic acid, aluminium chloride, or ferric chloride, which serves to break the dispersion and produce a finely-divided mechanical admixture of the asphalt.[216]

A self-hardening plastic cement has been proposed composed of finely ground hard asphalt (mixed with asbestos and fillers) and a softening agent (containing hard asphalt dissolved in a solvent, and containing paraffin wax and aluminium stearate), which are mixed together immediately before applying.[217]

BITUMINOUS VARNISHES

These correspond to the preceding classification, but carry a larger percentage of vegetable drying oil, with the fillers and pigments absent. In other words, they consist of a bituminous base, a volatile solvent, animal or vegetable "drying oils" with or without the presence of resins. The proportion of oils should be sufficient, so the base will possess a liquid to semi-liquid consistency and harden at room temperature by *oxidation* of the oils. *Hard* bituminous substances *only* are suitable for manufacturing varnishes, including the hard native asphalts, asphaltites, hard residual asphalts, wurtzilite asphalt, hard rosin pitch, hard wood-tar pitch,

hard fatty-acid pitch and hard bone-tar pitch. Those ordinarily employed include asphaltites, hard native asphalts, hard fatty-acid pitches, and less frequently wurtzilite asphalt. The oils generally employed include linseed and china-wood, or a mixture of the two.

Asphaltic varnishes are prepared in the same manner as oleo-resinous varnishes, namely, in a varnish kettle. When fossil resins (copals) are to be used, they are first melted and "depolymerized" at a temperature of 500 to 550° F., and when rendered fusible, the oils are added in the customary manner, then the dryers, and finally the asphalt or asphaltite.[218] When the mixture has been thoroughly melted, it is allowed to cool and thinned with one or more volatile solvents.

When the varnish consists solely of bituminous matter and drying oil, one of three procedures may be followed, viz.:

(1) The oil or mixture of oils in the raw state may be fluxed with the bituminous matter, thereupon "bodied" at 500 to 525° F. until the mass becomes stringy, cooled to 400 to 450° F., then the dryers incorporated, and finally thinned with a volatile solvent.

(2) The oil or mixture of oils may first be bodied alone, then fluxed with the bituminous matter, the heat raised to about 500° F., cooled to 400 to 450° F., the dryers incorporated, and finally thinned.

(3) The oil, or mixtures of oils may first be bodied as in the foregoing, cooled to 400 to 450° F., the dryers incorporated, fluxed with the bituminous matter, cooled and thinned.

Each varnish maker has his own views as to the relative efficiency of these procedures.

Varnishes made from linseed oil alone are more durable on exposure to the weather, but they dry slower than mixtures containing china-wood oil. When china-wood oil is used, it must be heated above 500° F., to prevent the varnish drying with a "flat" or "frosted" appearance, but it should never be heated *alone* above 500° F., otherwise it will gelatinize and become infusible. The presence of 30 per cent linseed oil, resins or bituminous matter, or a combination of these will *retard* gelatinization but not prevent it. China-wood oil mixtures containing 50 per cent of one or more of these substances may be heated without danger of gelatinization.

Bituminous varnishes ordinarily contain 15 to 20 gal. of oil per 100 lb. of bituminous matter, or mixtures of bituminous matter and resin. The durability of the varnish on exposure is in proportion to the quantity of oil present, so-called "long-oil varnishes" being more durable than "short-oil varnishes." Rosin esters have recently been used in combination with asphalts in this class of varnishes with good results, and prove fairly durable on exposure, also waterproof.

The following dryers are generally used:

(1) Lead dryers in amounts ranging from 2–4 per cent by weight of the oil. Since the asphaltic varnishes are dark in color, it is unnecessary to use "pale" dryers, and consequently litharge (PbO) or red lead (Pb_3O_4) will answer satisfactorily.

(2) Manganese dryers in percentages of 1–2 per cent, usually in the form of manganese dioxide (MnO_2) or manganese borate.

(3) Cobalt dryers in percentages of ¼–½ per cent of the oil, and added in the form of cobalt acetate (CoA_2).

The best results are obtained by using combinations of lead and manganese; manganese and cobalt; or lead, manganese and cobalt in approximately the ratios specified. The addition of prepared liquid dryers to the cold varnish will not answer in the case of asphaltic varnishes as with oleoresinous varnishes.

The following represent typical specifications for this type of varnish:[219]

Grade "A"—Normal Asphalt Varnish.

Appearance: Smooth and homogeneous; no livering or stringiness.

Color: Jet black.

Flash-point (Closed-cup): Not below 86° F.

Action With Linseed Oil: Varnish must mix readily to a homogeneous mixture with an equal volume of raw linseed oil.

Insoluble in Carbon Disulfide: Not more than 1 per cent.

Non-volatile Matter: Not less than 40 per cent by weight.

Fatty Matter: Not less than 20 per cent of the non-volatile. Must be liquid and not show a violet coloration by the Liebermann-Storch test.

Set to Touch: Within 5 hours.

Dry Hard and Tough: Within 24 hours.

Toughness: Film on metal must withstand rapid bending over a rod ⅛-in. in diameter.

Working Properties: Varnish must have good brushing, flowing, covering, and leveling properties.

Resistance to Water: Dried film must withstand cold water for 18 hours.

Resistance to Oil: Dried film must withstand lubricating oil for 6 hours.

Grade "B"—Asphalt Varnish Resistant to Mineral Acids.

Same as for Grade "A," but in addition must withstand acids, as follows:

Resistance to Mineral Acids: Dried film must withstand action of the following acids for 6 hours: sulfuric acid, sp. gr. 1.25 (about 33 per cent); nitric acid, sp. gr. 1.12 (about 20 per cent); hydrochloric acid, sp. gr. 1.09 (about 18 per cent).

Other specifications [220] have been proposed, similar to the foregoing, with the exception that the varnish is stated to contain not more than 0.95 per cent of mineral ingredients (i.e., manganese, lead, etc.).

Paracoumarone resin, produced by the polymerization of indene, coumarone, and their homologues, has been suggested for use in varnish in place of rosin-esters, hardened rosin, or the like. It is prepared from coal-tar naphtha distilling between 160 and 200° C. by the action of strong sulfuric acid or aluminium chloride. The resin is insoluble in alkalies, also alcohol, but will readily combine with china-wood oil and will retard its gelatinization, and will not darken when the mixture is heated to 320° C. Coal-tar naphtha should be used as solvent, and the product will mix readily with asphaltic preparations.[221] Another composition consists of a mixture of fatty-acid pitch with a phenol-formaldehyde resin dissolved in amyl acetate;[222] also a mixture of gilsonite, linseed oil, dryers, rubber, sulfur and a volatile solvent.[223] The following bituminous varnish is recommended for surfacing rubber compositions (e.g., footwear, etc.), being applied to the rubber before vulcanization: linseed oil is first air-blown until it attains a specific gravity at 60°/60° F. of 0.960-0.965, or else it is fire-cooked at 275° F. until it becomes thick and stringy; whereupon it is heated with 4-10 per cent of sulfur in a steam-jacketed kettle; next gilsonite, kapak, fossil resins, oil-black, or the like is incorporated; then dryers are added; and finally the mixture is thinned with petroleum naphtha.[224]

For surfacing fabrics coated with rubber or nitrocellulose lacquers, the following compositions are recommended: a mixture of asphalt, an alkyd resin, iron oleate, resinate or naphthenate, fillers and pigment;[225] or a baked coating consisting of asphalt, fatty-acid pitch, vegetable drying oil, pigment and thinner;[226] or a baked coating consisting of vulcanized asphalt flux oil, residual asphalt, vegetable drying oil, pigment and thinner;[227] or a baked coating composed of an asphaltite fluxed with a plasticizer (chlorinated diphenyl) and dissolved in turpentine.[228]

Bituminous varnishes will withstand exposure to the weather even better than oleo-resinous varnishes, although their use is limited by their inherently dark color, which restricts them to coating metal work either out-of-doors or in-doors. They produce lustrous coatings on drying. Other things being equal, the blacker the streak on porcelain of the bituminous base, the more intense will be the color, covering power and opacity of the resulting varnish. If too large a proportion of the resin or oil is used in its manufacture, the varnish will dry with a translucent film possessing a brownish-black to deep brown color, having little value for coating structural objects. Such varnishes are sometimes used by artists to produce warm brown tones, and were so employed by the ancient masters, who possessed the secret of preventing them diffusing through and discoloring the light-colored paints in juxtaposition.

BITUMINOUS ENAMELS

These are prepared from bituminous varnishes by grinding in dry pigments, sufficiently intense to overcome the inherently dark color of the varnish. The enamels at best can be manufactured in comparatively dark hues only, which limit their scope of usefulness. They have the advantages of extreme durability and low cost, since bituminous materials are less expensive than the resins otherwise used for this purpose.

The bituminous varnishes to be used for manufacturing enamels are made as translucent as possible to favor the color of the pigment. Bituminous substances showing a *brown* streak are accordingly selected, and combined with substantial amounts of resinous and oily bodies. Gilsonite[229] constitutes the most popular base, since it shows the optimum brown color, possesses the necessary

hardness, and at the same time does not tend to gelatinize with lead, zinc and chromium pigments.

The pigments are incorporated in the vehicle by grinding through a paint mill. The following pigments may be used:

Red iron oxide to produce deep reds and maroons.
Chrome green to produce deep shades of green and olive.
Yellow ochre to produce browns.
Chrome yellow to produce deep yellows and browns.
Lithopone, zinc white, white-lead, titanium oxide, and sublimed lead to produce tans (without other additions).
Graphite, used either alone, or in combination with lampblack or carbon black, for coating structural steel, and metal roofs.

The incorporation of aluminium stearate is claimed to retard the pigments from settling out of the enamel.[230]

CELLULOSE-ESTER LACQUERS

These lacquers are usually prepared by mixing together a solution of the bituminous constituents with a solution of cellulose ester, both of which are prepared separately, and the solvents so selected that neither will precipitate the solid constituents of the respective solutions. The cellulose-ester solution may be prepared from cellulose nitrate or cellulose acetate. If the former is employed, it should have a nitrogen content of 12.0 to 12.2 per cent by weight so as to provide a low viscosity when in solution. It is soluble in alcohols, esters and ketones, as well as mixtures of these. The best solvents for nitrocellulose include:[231]

Ethyl lactate (B. P. 155° C.) which dissolves completely rosin, rosin ester, shellac, kauri and pontianak copals. It mixes with hydrocarbon solvents and is not affected by the presence of water up to 25 per cent.

Diethyl carbonate (B. P. 125° C.) which dissolves completely rosin, rosin ester, mastic and cumar resins. The addition of 5 to 10 per cent of anhydrous ethyl alcohol effects a complete solution of sandarac, kauri and manila copal.

Diacetone alcohol (B. P. 164° C.) is not as good a solvent as ethyl lactate, but is a better solvent for congo copal.

Ethyl acetate (anhyd.—B. P. 77° C.) and butyl acetate (B. P. 120–140° C.) dissolve completely rosin, rosin ester, mastic and cumar resin. The addition of 5 to 30 per cent of anhydrous ethyl

alcohol effects a complete solution of elemi, damar and shellac, and a partial solution of sandarac, kauri, pontianak and manila copals.

The optimum ratio of nitrocellulose to resin is 2 : 1 by weight. Both are usually dissolved separately in suitable solvents and the two are then mixed. The resin may be wholly or partly replaced by glance pitch or gilsonite. Other forms of hard asphalt are sometimes used. These may be dissolved in amyl acetate, ethyl lactate, amyl chloride, chlor-acetic acid, or the like, and mixed with solutions of cellulose ester and resin respectively. A so-called "plasticizer" is generally added to the mixture to prevent the film from becoming brittle and to insure its adhesion to the under surface. Diamyl-phthalate (B. P. above 340° C.) is claimed to give the best results and may be present up to 50 to 60 per cent of the nitrocellulose content. Castor oil is sometimes used for the same purpose. Other plasticizers include: camphor (M. P. 180° C., B. P. 209° C.); acetanilid (M. P. 115° C., B. P. 304° C.); di-ethyl phthalate (B. P. 170–176° C. at 20 mm., F. P. 140° C.); di-butyl phthalate (B. P. 200–214° C. at 20 mm., F. P. 160° C.); di-isobutyl phthalate (B. P. 191–200° C. at 20 mm., F. P. 161° C.); di-methyl phthalate (B. P. above 340° C.); di-amyl phthalate; tri-cresyl phosphate (B. P. 340° C., F. P. 233° C.);[232] tri-phenyl phosphate (M. P. 53° C., B. P. 320° C., F. P. 235° C.); tolyl phosphate; ethyl abietate; etc. The use of esters of cyclohexanol, or the corresponding ketones, as solvent will facilitate the incorporation of cellulose-ester lacquers with bituminous-solvent compositions.[233]

Where colors are to be obtained, various colored pigments may be added to the mixture, up to 25 or 30 per cent by weight. Lacquers of this type dry quite rapidly to a semi-glossy coating, possessing an unusual degree of toughness, and will withstand atmospheric exposures very well.

Many combinations involving the use of cellulose esters have been patented.[234] These also include asphalts, asphaltites, nitro derivatives of asphaltites[235] (which have the advantage of being soluble in ethyl alcohol, acetone, and the most of the cellulose-ester solvents), fatty-acid pitch,[236] chlorinated asphalts,[237] wood-tar pitch,[238] paraffin wax,[239] etc.

Typical mixtures include the following: coal-tar pitch or petroleum asphalt or gilsonite, coumarone resin or rosin ester, nitrocellu-

lose, tri-cresyl phosphate, butyl acetate, toluol or xylol; coal-tar pitch or petroleum asphalt or gilsonite, nitrocellulose or ethyl-cellulose, blown castor oil, tri-cresyl phosphate, ethyl or butyl acetate, benzol, toluol or xylol, with or without resins (e.g., copal) and colored pigments; [240] a mixture of pitch, phenol and benzol; [241] a mixture of asphalt, coumarone resin and solvent; [242] a mixture of asphalt, fatty-acid pitch, celluloid waste and solvent; [243] etc. The addition of cellulose-acetate lacquers to aqueous asphalt dispersions has also been suggested.[244]

BITUMINOUS JAPANS

In this treatise the term "japan" is used to designate a dark-colored menstruum applied to metals, wood, fabrics, etc., intended to be hardened by *baking*. Japans are sometimes used for impregnating felted or woven fabrics for electrical insulating purposes. In composition they fall into any of the four groups already enumerated, the fillers and pigments being omitted. The base of cheap japans is composed solely of bituminous materials blended in the proper proportions, whereas the better grades are made from mixtures of bituminous materials and vegetable drying oils, with or without the addition of resin. They are opaque black to translucent brown in color, and after baking, form coatings which are extremely hard, tough and resistant to abrasion.

Japans are often made from asphaltites (e.g., gilsonite), semi-solid native asphalts, and in some cases residual asphalts derived from aromatic-base petroleum which have the property of hardening on "baking." Toughness may be imparted by fluxing with a small percentage of blown petroleum asphalt or fatty-acid pitch. Fatty-acid pitches, which have been overheated in their process of manufacture, are especially suitable for this purpose, being often used alone. The characteristics of bituminous substances suitable for manufacturing japans are as follows:

(1) They must be homogeneous and free from any fine particles of grit.

(2) They should show a black streak, and be opaque when viewed in a thin layer.

(3) They must not separate, curdle or "gelatinize" on thinning with petroleum naphtha.

(4) They must bake in a reasonable length of time to a tough and permanently glossy coating, without shrivelling or "crazing."

A popular form of japan consists of gilsonite fluxed with a fatty-acid pitch of the type described, and thinned with petroleum naphtha to brushing consistency. If the fatty-acid pitch does not bake sufficiently rapid, a common procedure consists in combining a small percentage of dryer, such as manganese dioxide or raw umber, at a temperature of 450° F. Tougher and more elastic japans are prepared by incorporating a proportion of bodied linseed oil, which also serves to improve the gloss.[245]

Since semi-drying vegetable and animal oils will oxidize to tough coatings at elevated temperatures, and are usually less expensive, they may be substituted for the vegetable drying oils, especially if bodied and combined with dryers. Thus cottonseed oil, blown castor oil,[246] corn oil, soya bean oil, and fish oil [247] have been used for this purpose, sometimes combined with a small percentage of china-wood oil to accelerate the drying. Another expedient consists in bodying the vegetable oils at 450–475° F. with a small proportion of Prussian blue (½–1 ounce per gallon), which serves to convert the oil into a brownish-black liquid, thus increasing the opacity of the japan, and at the same time imparting drying properties to the oil. This is the same procedure followed in manufacturing baking varnishes and japans for the patent-leather industry and in the manufacture of leather substitutes.[248] The addition of iron oleate or resinate produces similar results.[249]

Japans are applied to articles by dipping, brushing or spraying, and heated in specially constructed ovens from one to four hours at 200–400° F., depending upon their composition. Modern practice favors the use of higher temperatures for shorter periods of time. Wooden and other porous articles must be baked longer than metal. Semi-glossy and flat black japans are prepared by grinding carbon or lampblack in the foregoing menstrua, and when baked present a surface simulating the appearance of hard rubber. Sometimes a small percentage of oxide of zinc is incorporated to impart a grayish cast. Baked japans form a harder, tougher and more weatherproof coating than those resulting from bituminous varnishes allowed to air-dry at room temperature.[250]

Japans containing 10–17 gal. of drying oil per 100 lb. of solid constituents are employed for insulating armature and field coils of motors and dynamos, also transformer and magnet coils, in small

plants where vacuum impregnating apparatus is not available.[251] The coils after having been wound with cotton or other fibrous material are heated in an oven until the moisture has been expelled, and while still hot are dipped into the cold japan. The drying may be hastened if the fibrous covering is first preheated in ozonized air.[252] After removing and allowing the excess to drain, the coils are baked in an oven at 180–200° F., usually from 8–12 hours. It is advisable to dip and bake the coils twice to insure thorough impregnation. These japans are sold under the trade name of "black baking varnish," and if properly prepared will withstand an alternating current having a potential of 800–1000 volts per mil, after having been baked 300 hours. One of the essential characteristics of the "varnish" is that it shall neither soften nor become brittle when applied to armatures of dynamos or motors subjected to continual vibration, and often becoming heated as high as 190° F. in service. Should the varnish or japan become brittle, the vibration will soon cause it to crack and pulverize, and result in short-circuiting the machine. This is one of the severest tests to which any form of varnish is subjected. It requires considerable skill to prepare a japan that will retain its pliability on long service when subjected to alternate heating and cooling in contact with air, combined with the vibrating and centrifugal action exerted by the revolving armature. A simple test consists in baking the japan on a small strip of muslin at 195° F. for 300 hours, and then observing whether it may be bent double (flat on itself) without cracking at 77° F. It must also withstand continuous immersion in hot lubricating oil without softening or disintegrating.

Resins are sometimes added to the baking japans, but are not looked upon with favor because of their tendency to make the coating brittle. It is claimed, however, that a mixture of phenol-formaldehyde condensation product and fatty-acid pitch dissolved in amyl acetate will resist heat without becoming brittle, better than linseed oil preparations.[253] Virtue is also claimed for a mixture consisting of asphalt or an asphaltite dissolved in a solvent and heated with a small amount of copper sulfate in an autoclave at 5 to 8 atmospheres.[254] Wrinkle-finish baking-japans have also been described.[255]

Emulsion japans, known to the trade as "water japans" are recently meeting with much favor. They consist of the same ingredi-

ents which have already been described, in which however, water takes the place of the volatile solvent. Any of the dispersing agents previously described may be used for the purpose.[256] An alternate process consists in treating linseed oil with alkali to form a soap, evaporating the water, melting the residue with gilsonite, cooling to 150° C., running in boiling water with stirring, and then quickly cooling to room temperature.[257] The addition of an alkaline solution of sugar insures stabilization of the emulsion, and causes the japan to yield a mat surface on baking.[258] A mat surface may also be produced by treating the baked japan with concentrated sulfuric acid and then washing with water.[259]

PART VI

METHODS OF TESTING

CHAPTER XXXI

SAMPLING

The methods which follow are suitable for sampling the following classes of bituminous materials:

(A) Crude, Refined and Blended Bituminous Substances.
(B) Bituminized Mineral Aggregates.
(C) Bituminized Fabrics.
(D) Bituminous Lacquers, Cements, Varnishes and Japans; also Bituminous Emulsions.

For sampling purposes, bituminous materials may be classified according to their physical condition, as follows:

(1) Liquids to semi-liquids.
(2) Highly viscous substances.
(3) Coherent semi-solids to solids.
(4) Lumpy solids in crushed fragments or powdered form.
(5) Sheet or roll form.

These methods apply to material in the following types of containers:

(*a*) Cans or small containers (up to 10 U. S. gal., inclusive); (*b*) Drums of barrels; (*c*) Tank cars or tank trucks; (*d*) Shore tanks; (*e*) Ship tanks or barge tanks; (*f*) Pipe lines; (*g*) Bags; (*h*) Cakes; (*i*) Bulk or loose form.

Samples are taken for the following types of tests:

(*a*) General quality tests; (*b*) Definite chemical and physical tests; (*c*) Special quality tests.

Samples are used for one or more of the following purposes:

(*a*) Visual examination; (*b*) Laboratory test; (*c*) Preservation for record; (*d*) Check and referee tests; (*e*) The extent of variation of the quality in different portions of the lot; (*f*) The average quality of the whole lot of material.

Samples should be marked, recorded, and handled throughout in accordance with all the usual precautions of the best laboratory technique.

DEFINITIONS [1]

*Average Sample.** An average sample would be one so taken as to contain parts from all sections of the container or pipe in proportion to the volume of each part.

All-levels Sample.† An all-levels sample is one obtained by submerging a closed sample container to a point as near as possible to the draw-off level, opening it and raising it at such a rate that it will be nearly but not quite full when withdrawn.

Upper Sample. An upper sample is one taken at a point 10 per cent of the depth of a uniform cross-section vessel or 10 per cent of the diameter of the horizontal cylindrical tank below the surface.

Middle Sample. A middle sample is one taken at half the depth of the material.

Lower Sample. A lower sample is one taken at a point 10 per cent of the depth of a uniform cross-section vessel or at 10 per cent of the diameter of a horizontal cylindrical tank above the bottom of the vessel.

Composite Sample. A composite sample is a mixture of upper, middle and lower samples containing, for vessels of different shapes,

* It is practically impossible to obtain an average sample, except, perhaps, through a continuous sampling connection from a vertical run in a pipe line with specially constructed draw-off pipes, or by vigorously agitating and stirring the contents of a vessel and drawing off a sample while the contents are still agitated. Samples obtained by lowering and raising a bottle while allowing it to fill have been considered average samples, but obviously the proportions are not related to the tank volumes at the various levels.

† The all-levels sampling method is widely used for ship tanks, barges and large shore tanks. The method can not, however, give an average or a strictly representative sample, not only because the tank volume may not be proportional to the depth and because the operator can not move the sample container with a uniform speed, but also because the rate of filling the sample container is proportionate to the square root of the depth of immersion of the sample container opening below the surface.

volume proportions which correspond approximately to the volumes of the material at these levels, as follows:

	Composite Sample Proportions	
	Uniform Cross-Section Vessels	Horizontal Cylindrical Tanks (Assumed Full)
Upper sample	1 part	1 part
Middle sample	3 parts	8 parts
Lower sample	1 part	1 part

For horizontal cylindrical tanks that are only partially filled, the simple geometrically proportioned sampling levels, and composite sample mixtures stated above cannot give samples that are substantially proportionate to the volumes at the different depths, unless the tanks are filled to at least 80 per cent of the diameter.

In referee tests, a mutually satisfactory selection of sampling levels and sample quantities at each level shall be mutually agreed upon, if any departure from the methods specified is to be allowed.

Table LXXIX gives a set of substantially correct sampling levels and sample quantities for partially filled horizontal cylindrical tanks, which may be used in the absence of other arrangements, em-

TABLE LXXIX

SAMPLING LEVELS AND SAMPLE QUANTITIES FOR PARTIALLY FILLED HORIZONTAL CYLINDRICAL TANKS

Liquid Depth, Percentage of Diameter	Sampling Level, Percentage of Diameter above Bottom			Quantity of Sample to be Taken at Each Sampling Level		
	Upper	Middle	Lower	Upper	Middle	Lower
10	.....	.....	5	.....	.....	10 parts
20	.....	.....	10	.....	.....	10 parts
30	.....	20	10	.....	6 parts	4 parts
40	.....	25	10	.....	7 parts	3 parts
50	.....	30	10	.....	8 parts	2 parts
60	55	35	10	1 part	8 parts	1 part
70	65	40	10	1 part	8 parts	1 part
80	75	45	10	1 part	8 parts	1 part
90	85	50	10	1 part	8 parts	1 part
100	90	50	10	1 part	8 parts	1 part

ploying values in the tabulation that lie nearest to the *actual filling depth.*

Continuous Sample. A continuous sample is one obtained from a pipe or conduit conveying the material, in such a manner as to give at all times a representative average of all the sections and section velocities of the stream throughout the period of transit.

Dipper Sample. A dipper sample is one taken by interposing a dipper or collecting vessel into the path of a free flowing stream, so as to collect a definite volume from the full cross-section of the stream at regular time intervals, or at time intervals so varied as to obtain samples in proportion to the amounts being delivered.

Mixed Sample. A mixed sample is one obtained by mixing or vigorously stirring the contents of the original contained and pouring or drawing off the quantity desired.

Thief Sample. A thief sample is one taken by means of a sampling tube from a specified point in the containing vessel being sampled.

*Drain Sample.** A drain sample is one taken by opening the draw-off or the discharge valve and drawing off a suitable portion of the material.

*Bottom Sample.** A bottom sample is one obtained by collecting a portion of the material lying on the bottom surface of the tank, container or line at its lowest point.

Borings Sample. A borings sample is one obtained by collecting the chips made by boring holes with a ship-auger from top to bottom of the material contained in a barrel, case, bag, or cake.

Grab Sample. A grab sample is one obtained by collecting representative portions from loose solids in equal quantities from each part or package of a shipment, in sufficient amount to be representative of all the sizes and components.

GENERAL DIRECTIONS AND PRECAUTIONS

Official samples shall be taken by, or under the immediate supervision of, a person of judgment, skill and previous experience in sampling. The portions taken for samples shall represent the

* Occasionally, as in tank cars, the drain sample will also give a bottom sample. Drain samples and bottom samples are usually taken only in order to examine for moisture, sludge, scale, etc., and in some cases for the purpose of drawing off any free water or extraneous material.

general character and average condition of the lot sampled. If there is any substantial reason indicating that a sample or set of samples may not be fully representative, another sample or set of samples shall be taken.

Great care and good judgment are necessary in taking and handling samples from crude or unrefined materials, not only because of possible segregation, sludging and volatilization, but because of the bearing upon value, refining problems, and methods of handling. A high degree of care shall be employed in handling samples from semi-refined materials upon which definite chemical and physical tests are usually made. The utmost precautions and the most meticulous care shall be exercised in handling samples from refined products, upon which special quality tests are usually made.

For all grades of materials, precautions shall be taken to assure that the sampling apparatus and the samples themselves are neither contaminated with *nor altered by any material* not representative of the lot being sampled.

The operator engaged in sampling shall have clean hands, free from any material (unless it be the material being sampled). Cleaned gloves may be worn, but only when absolutely necessary, as in extremely cold weather, or in handling materials at high temperatures, or materials involving some health or other hazard.

The sampling apparatus shall be thoroughly clean and dry, and free from any substance that will dissolve into and/or color water-white gasoline.

NOTE.—*Exception:* Crude and dark-colored semirefined materials (such as dark-colored fuel oils) when fluid enough to drain completely from sampling apparatus (Saybolt universal viscosity less than 250 at 100° F.) may be sampled repeatedly with apparatus that has been hung up and thoroughly drained.

Samples shall be permanently marked (preferably with a pencil hard enough to dent the paper)* immediately after collection, with the date, also the hour and minute for continuous and dipper samples, name of the sampler, name or number and owner of vessel, barge, car or container; material sampled, and a reference symbol or number. Thin aluminium strips stamped with the sample number and other information are acceptable. The reference symbol or

* Soft pencil and ink markings are liable to obliteration from moisture, handling or smearing with oil.

number and other markings should be entered in a bound record book.

Sample containers shall be closed immediately after collection. Sample containers shall be of such types as will protect the sample from contamination or deterioration. Specific precautions shall be taken as follows:

(*a*) Corks, for bottles, shall be clean, free from holes and loose bits of cork.

(*b*) Sealing wax, or paraffin, shall *not* be poured over corks after filling.

(*c*) Light-sensitive samples, when placed in bottles, shall be wrapped or otherwise covered at once, to protect them from light.

(*d*) Tin cans with screwed or overlapped and soldered caps are acceptable, but only if the inside has been scrupulously cleaned and known to be free from dirt, water, washing compounds, naphtha or other solvents, soldering flux or acids, corrosion, rust, and pin holes.

(*e*) Refined materials should be further protected by covers of paper or metal-foil over the stopper and top to keep moisture and dust away from the filling and emptying opening, and to protect it from contact with the hands. Covers also prevent sealing substances, such as wax or paraffin, from getting on the lip when the seal is made or later opened, and so getting into the sample when it is poured out.

(*f*) Samples shipped by mail, express or messenger shall be well packed to avoid spillage, leakage, breakage, and loss by evaporation or alteration while in transit.

Volatile samples shall be protected from evaporation while being collected, particularly in the case of continuous and dipper samples, and at once placed in closed containers. Samples shall, if possible, be taken at the time of loading of a shipment and at the point of origination of a shipment. When sampling at the point of origin has not been provided for, or is not feasible, then the sampling shall be carried out as soon as possible after receipt of the shipment.

(A) SAMPLING CRUDE, REFINED AND BLENDED BITUMINOUS SUBSTANCES

The following procedure [2] is recommended for sampling all types of bituminous substances in various physical conditions, with the exception of bituminized fabrics in sheet or roll form, for which special methods have been devised as will be described later.

Care shall be taken that the samples are not contaminated with dirt or any other extraneous matter and that the sampler containers are perfectly clean and dry before filling. Immediately after filling, the sampler containers shall be tightly closed and properly marked for identification on the container itself or on a linen tag attached to the container.

(*a*) Whenever practicable, bituminous materials shall be sampled at the point of manufacture, and at such time as to allow the tests, controlling acceptance or rejection, to be made in advance of shipment.

(*b*) When impracticable to take samples at the point of manufacture, they should be taken from the shipment immediately upon delivery.

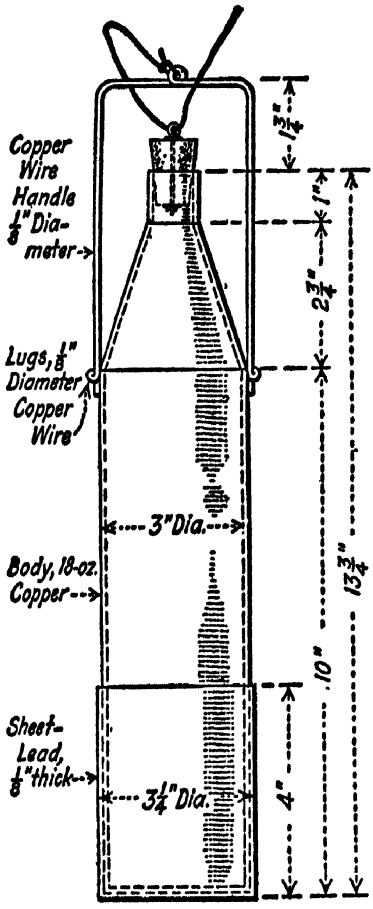
For routine laboratory examination of acceptability of a given lot, not less than one quart of material should be submitted, which should be representative of the average sample collected as hereinafter described. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps. Containers for semi-solid and solid materials shall be friction-top cans.*

Sampling at Place of Manufacture

The inlet and outlet to the storage tank shall be sealed and a 1-gal. sample drawn from the top, middle and bottom contents. The sample may be taken from drain cocks on the side of the tank, if such are available. Enough material should be discarded to insure a representative sample. Otherwise, samples may be taken by lowering weighted bottles or beakers into the material.[3] The sampling bottle and sampling beaker shall be made substantially in accordance with the form and dimensions shown in Fig. 193, and the following instructions shall apply to a substantially full tank:

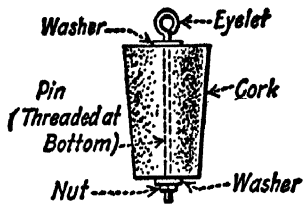
(*a*) An upper sample shall be taken by lowering the weighted stoppered bottle or beaker till its mouth is 10 per cent of the depth of a tank of uniform cross-section or diameter of a horizontal cylindrical tank below the surface, uncorking it by a quick jerk, allowing it to fill completely, as evidenced by cessation of air bubbles, and then immediately withdrawing it.

* Semi-solid to solid bituminous materials when sampled may be conveniently transported to the laboratory in tin cans or boxes, the inside of which has been amalgamated with mercury, to which they do not adhere.

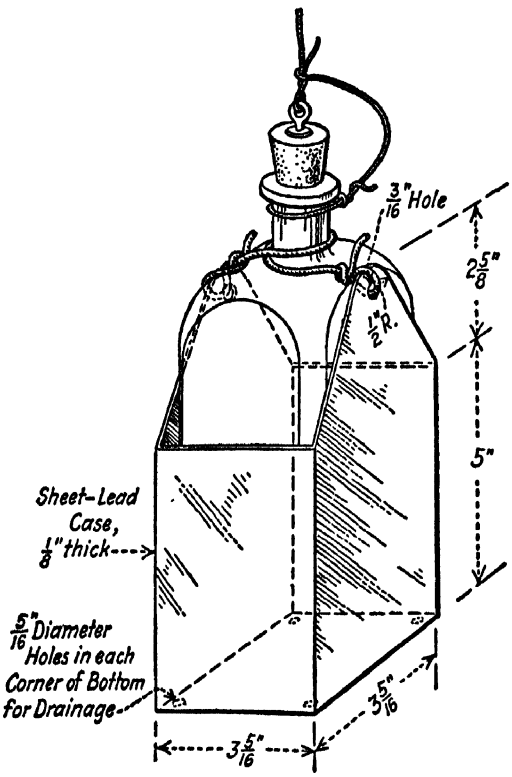


Weighted Beaker.

Note: This Container Should be Used when one Composite Sample is desired for each Grade of Cargo.



Detail of Cork Arrangement.



Weighted Bottle.

Note: This Bottle to be used for Obtaining the Individual Samples when Individual Samples are to be kept Separate, that is whenever the Sample must be delivered to the Laboratory in the same Container in which it was contained.

Courtesy A.S.T.M.

FIG. 193.—Standard Sampling Containers for Bottle or Beaker Sampling.

(*b*) Three middle samples shall be taken in a similar manner from a tank of uniform cross-section and eight middle samples from a horizontal cylindrical tank with the bottle or beaker mouth lowered to the middle of the tank.

(*c*) A lower sample shall be taken in a similar manner with the bottle or beaker mouth lowered to a point 10 per cent of the depth of a tank of uniform cross-section or diameter of a horizontal cylindrical tank above the bottom of the tank.

(*d*) The upper, middle, and lower samples from each tank car, tank truck, shore tank, individual ship tank or barge tank shall be combined into a composite sample.

(*e*) The composite samples taken with the beaker from the various ship tanks shall be combined into a mixed cargo sample.

(*f*) The beaker samples shall be poured as taken into a clean 5 or 10-gal. can, or cans, and the cans closed and covered, labeled and delivered to the laboratory, shaken or mixed with the churn-dasher type of stirrer, and portions withdrawn by means of a thief for test.

(*g*) The composite samples taken with the bottle from the various ship tanks shall be kept separate and delivered separately to the laboratory.

(*h*) The bottle samples shall all be delivered to the laboratory in the bottle in which they were taken.

(*i*) When loading or discharging any finished product, samples shall also be taken from the shore tanks and at frequent intervals from the shore pipe line on the dock. These samples shall also be tested whenever it seems advisable.

(*j*) A portion of all samples from all shipments shall be fully labeled and kept in storage until the final disposition of the material.

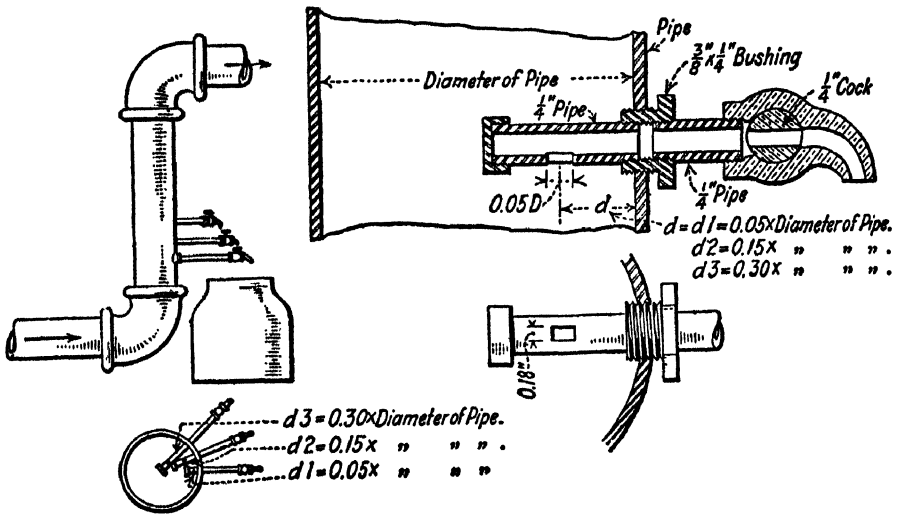
(*k*) Ship and barge tanks should be sampled not only immediately after loading, but also before discharging.

Where tank cars, distributors or barrels are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as hereinafter described.

When Material is Pumped under Pressure. In the case of pipe lines, filling lines and transfer lines, the continuous method of sampling [4] is applicable. The continuous sampling connection shall be constructed and installed in accordance with the form and dimensions shown in Fig. 194, and the churn-dasher form a stirrer as shown in Fig. 195. The three plug-cocks shall be opened, as nearly as possible, to the same angle of opening, so that a steady stream is drawn off from each cock and at such a rate as will allow about 0.1

per cent of the stream to be diverted during the passage of the lot being sampled, but not more than 40 U. S. gal. The following precautions should be observed:

(*a*) For semi-liquid materials, the receiver, or receivers, shall be kept warm by means of a steam-coil adjusted to keep the material just above the liquefying temperature.

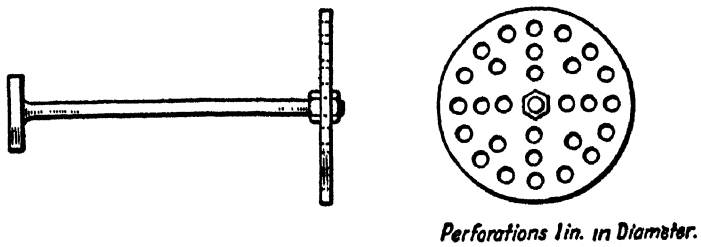


Courtesy A.S.T.M.

FIG. 194.—Continuous Sampling Connection.

(*b*) The receiver, or receivers, should be kept covered and closed, except for their vents and filling stream openings, or pipes.

(*c*) Screens of larger area than the openings may be placed



Courtesy A.S.T.M.

FIG. 195.—Churn Dasher Type of Stirrer.

around the ¼-in. pipes, or ¼-in. screen fittings inserted in the drip lines.

(*d*) A pipe-cap with a suitable orifice hole may be substituted for the cocks, where the orifice size has been predetermined so as to give the right amount of sample. Care should be taken that the

drip-cock, or pipe-cap, after once having been set, allows a constant flow during the pumping.

(*e*) The gross continuous sample as collected shall be stirred with the churn-dasher type of stirrer, or mixed by rolling or shaking, and then sampled by means of a thief to get a 1-quart sample, or more where larger quantities are called for. This method is also applicable to gravity flows where the pipe line is completely filled by the outflowing liquid and has a rising section.

When Materials Flow by Gravity. Materials flowing by gravity through pipe lines which are not completely filled may be sampled by taking dipperfuls [5] at the outlet at frequent and regular intervals. The dipper shall have a flared bowl holding approximately 1-quart and a handle of any convenient length. Tinned steel is acceptable. The procedure shall be as follows:

(*a*) The dipper shall be inserted in the free flowing part of the stream, collecting a sample from the full cross-section of the stream at regular intervals, and the sample collected poured into a clean can of a convenient size, such as 5- or 10-gal. capacity.

(*b*) If the pumping or delivery rate varies, the samples shall be taken at time intervals, so chosen as to give a sample for equal pumped quantities.

(*c*) The container into which the individual dipper samples of approximately 1-quart each are poured shall be kept closed and covered, except when the close-fitting lid and cover are lifted to pour in the dipper sample.

(*d*) The gross amount of material collected should be about 0.1 per cent of the quantity transferred, and the time intervals should be so chosen as to correspond, but not less than 5 gal. nor more than 40 gal. should be collected.

(*e*) The 5- or 10-gal. can shall be closed and covered, labeled and delivered to the laboratory, where it shall be shaken or mixed with the churn-dasher type of stirrer and portions withdrawn by means of a thief for test.

Sampling at Point of Delivery

Samples may be taken by means of a weighted bottle or can, or from the unloading pipe line as described above. Liquid bituminous materials shall be sampled before heating. Semi-solid or solid bituminous materials shall be rendered fluid by heating. Sampling should be so conducted as to eliminate the possibility of adventitious water resulting from leaky steam heating coils, rain or snow.

Samples may be taken from distributors by means of a weighted bottle or can, as described. One sample shall be sufficient.

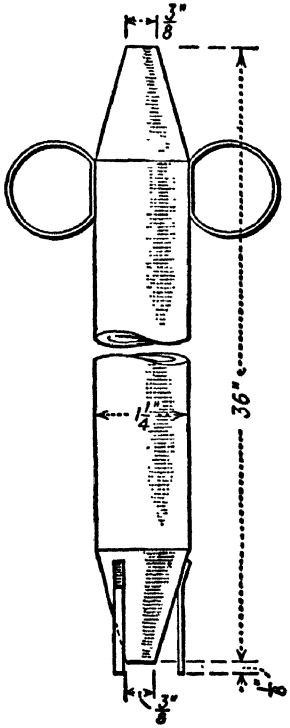
(*a*) *Semi-solid or Solid Materials.* Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package or cake shall be selected at random and sampled as described in the following paragraph. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single sample selected as described above fails on test to conform to the requirements of the specifications, a number of packages or cakes shall be selected at random equivalent to the cube root of the total number of packages or cakes in the lot. For convenience, Table LXXX is given, showing the number of samples to be selected for shipments of various sizes:

TABLE LXXX

Packages or Cakes in Shipment	Packages or Cakes Selected	Packages or Cakes in Shipment	Packages or Cakes Selected
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1331	11

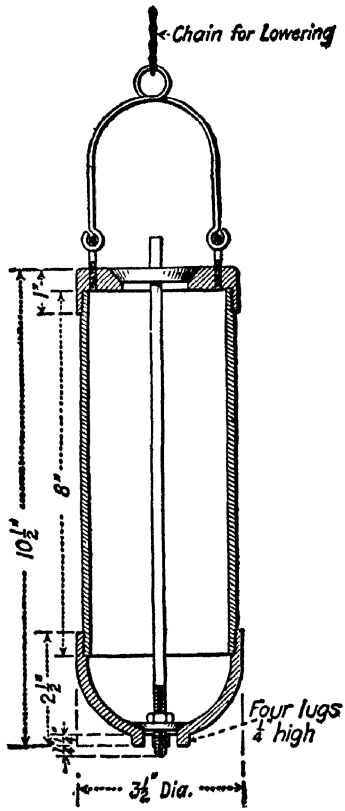
Samples shall be taken from at least 3 in. below the surface and at least 3 in. from the side of the container or cake, or from the center of a cake. A clean hatchet may be used if the material is hard enough to shatter and a broad stiff putty knife if the material is soft. An auger, or brace and ¾-in. bit, or other suitable means may also be used. When more than one package or cake in a lot is sampled, each individual sample shall be not less than ¼ lb. in weight. When the lot of material is obviously from a single run or batch of the producer, all samples from the lot shall be melted and thoroughly mixed, and an average sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and the batches can be clearly differentiated, a composite sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches, each sample shall be examined separately.

(*b*) *Liquid Materials.* Samples of liquid bituminous materials from cans, drums or barrels, or tank-cars may be taken by the so-called "thief sampling" method.[6] For sampling drums, the thief shall be so designed that it will reach within approximately 1/8-in. of the bottom and should have a capacity of approximately 1-pint or 1-quart. A convenient thief for sampling 50-gal. drum contain-



Courtesy A.S.T.M.

FIG. 196.—Thief for Sampling Drums.

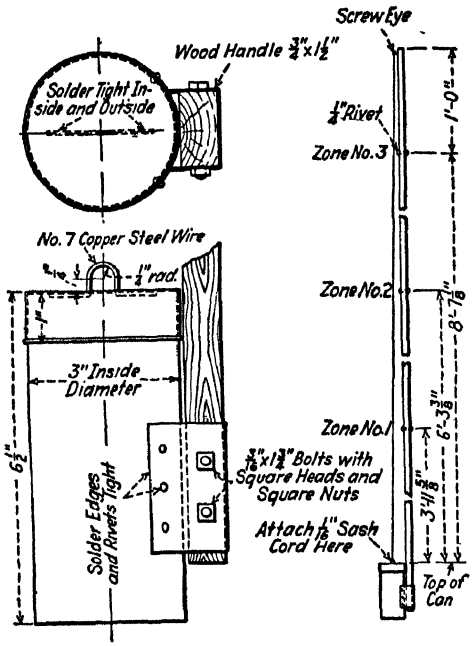


Courtesy A.S.T.M.

FIG. 197.—Thief for Sampling Tank Cars.

ers is shown in Fig. 196. Three legs, equally spaced around the thief at the lower end, long enough to hold the opening 1/8-in. from the bottom of the container, aid in securing a good representative sample. Two rings soldered to opposite sides of the tube at the upper end will be found convenient for holding the thief by slipping two fingers through them, leaving the thumb free to close the opening. For sampling cans of 5-gal. capacity or larger, a thief

similar to that used for sampling drums but of proportionately smaller dimensions shall be used. For sampling tank-cars, the thief shall be so designed that the sample may be obtained from within at least 0.5-in. of the bottom. One type of thief suitable for sampling tank-cars is illustrated in Fig. 197, being made from metal tubing and castings, with a valve rod, whose projecting stem strikes the bottom of the car, opening the valve automatically and simultaneously releases the air through the top. Another type of thief designed specially for sampling tank-cars of creosote oil as illustrated in Fig. 198, consists of a wide-mouthed vessel attached to a wooden rod fitted with a lid that can be withdrawn by means of a cord. This device shall first be lowered until the point marked "Zone No. 1" on the rod is level with the top of the dome of the tank car. The cover of the sampling can shall then be removed, the vessel allowed to fill at that point and then slowly withdrawn. Extreme care shall be exercised in lifting the rod so as not to disturb the contents of the sampling vessel. Two samples shall be taken in a similar manner with the sampling apparatus lowered to the point marked "Zone No. 2," and one sample shall be likewise taken with the sampling apparatus lowered to the point marked "Zone No. 3." The four samples shall then be combined and mixed at once into a composite sample while the material is thoroughly liquid.



Courtesy A.S.T.M.

FIG. 198.—Zone Sampling Apparatus for Creosote Oil.

Samples of liquid materials taken from cans, drums or barrels shall be secured by taking 1 qt. of material from packages selected at random according to the cube root method described in section (*a*). When the lot of material is obviously from a single run or batch of the producer, the samples shall be combined and thor-

oughly mixed and an average sample taken from the combined material. In case more than one run or batch is present and can be clearly differentiated, a composite sample shall be prepared from each batch. Where it is impossible to differentiate between the various batches or runs, each sample shall be examined separately.

(*c*) *Solid Bituminous Materials in Crushed Fragments or Powder.* Materials such as gilsonite, grahamite, etc., shall be sampled in accordance with methods for sampling coal,[7] or for less accurate work by the method of "grab sampling." [8] This method is applicable to all lumpy materials loose in bins, bunkers, barrels, boxes, or contained in sacks. A shovel, if employed for sampling, may be of any convenient size, but should have a width at least twice as great as the maximum dimension of the largest pieces sampled. Grab-sampling is effected by the following general procedure:

Solids are almost always heterogeneous in constitution. It is impossible to give absolutely definite and arbitrary methods for obtaining samples. Each problem must be worked out for itself, bearing in mind the particular conditions.

It is usually preferable to take samples during the unloading of cars or during transit of the material in conveyors. In such instances, a number of small samples should be taken at frequent and regular intervals from the material in transit and these samples combined to form a representative combined sample.

Occasionally, solids are tested as received in bags or barrels. In such instances, it is desirable to take a small sample from a number of packages selected at random and equivalent in number to the cube root (or the next larger whole number), of the total number of packages in the lot.

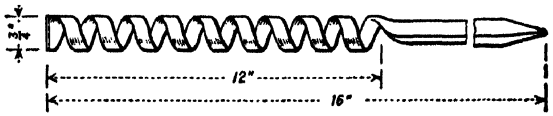
Generally, samples taken from the bulk, in piles or cars, are unreliable and not representative. Where it is necessary to take such samples before the unloading of the car, small samples should preferably be taken from at least twelve spots throughout the bulk and these small samples collected to form the representative combined sample. In taking such small samples, it is desirable to take eight samples from the corners of the car, four near the bottom and four near the top of the material. To those should be added four samples from the center of the car, two at the top, and two near the bottom of the material.

The combined sample taken by any of the above methods should be in amount at least 0.1 per cent of the total bulk of material sampled. These combined samples should be carefully mixed and reduced in size to a convenient laboratory sample, by the standard

method of quartering. In carrying out this quartering, a hard clean surface should be selected, free from cracks and protected from rain, snow, wind and beating sun. Cinders, sand, chips from the floor or any other material shall be avoided. The sample shall be protected from loss or gain in moisture or dust. The combined sample should be carefully mixed, spread out on the surface into a circular layer and divided into four equal quadrants. Two opposite quadrants shall be combined to form the representative reduced sample. If this sample is still too large for laboratory purposes, the quartering operation shall be repeated. In this manner, the sample shall finally be reduced to a size suitable for handling in the laboratory.

For wax and other soft solids in cases, cakes, bags or barrels, when they cannot conveniently be melted and sampled as liquids, a method of "borings sampling" [9] is recommended, as follows:

Use a ship auger which shall be ¾-in. in diameter and shall conform to the form and dimensions shown in Fig. 199, and be of such a length as to pass entirely through the material to be sampled.



Note. Greater Overall Lengths may be obtained when necessary to pass entirely through the Material to be sampled.

Courtesy A.S.T.M.

FIG. 199.—Auger for Sampling Soft Solids.

Opening: Cases and barrels shall have the covers or heads removed. Bags shall have the mouths opened. Cakes shall have the wrappings, if any, opened.

Foreign Matter: If foreign substances, such as dirt, sticks, string, etc., appear upon the surface, they shall be removed. Foreign matter found in the interior shall be included, as it may come, with the borings.

Boring Hole Location: Three test holes shall be bored through the body of the material as follows:

One at the center; one at one-quarter of the diameter (or width of the package) from the right side; one at one-quarter of the diameter (or width of the package) from the left side.

If any visible differences appear in the three sets of borings, they shall be preserved, examined and tested separately. If no visible

differences appear in the borings they may be combined, preserved, examined and tested as a single sample, but this shall be done only by the laboratory tester after the three sets of borings have been delivered.

Sub-division: If sub-division of the borings is desired, they may be chilled, pulverized if necessary for handling, mixed and quartered till reduced to the amount desired, after their receipt in the laboratory. This sub-division shall not be done in the field by the sampler.

(B) SAMPLING BITUMINOUS PAVING MATERIALS

The special methods given in Table LXXXII are recommended for sampling paving materials.[10]

C) SAMPLING BITUMINIZED FABRICS

The following procedure [11] should be adopted in sampling bituminized fabrics put up in the form of rolls or bundles:

From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, Table LXXXI is given, showing the number of samples to be selected from shipments of various sizes:

TABLE LXXXI

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 1000	5	5,833 to 8,000	10
1001 to 1728	6	8,001 to 10,648	11
1729 to 2744	7	10,649 to 13,824	12
2745 to 4096	8	13,825 to 17,576	13
4097 to 5832	9	17,577 to 21,952	14

TABLE LXXXII

Sampling Bituminous Paving Materials

Material	Method	Quantity	When Collected	By Whom Tested
Asphalt Sand	Standard methods of sampling stone, slag, gravel, sand and stone block for use as highway materials, including some material survey methods (A.S.T.M. designation: D 75).	5 lb.	Each car or boat and truck deliveries by special instructions from preliminary or first shipments from given source or when source or supply changes or if change occurs in materials received.	Laboratory.
			Daily from subsequent shipments, and sand in use from stock pile.	Plant Laboratory.
Screenings	Same as for asphalt sand.	5 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Crushed Stone	Same as for asphalt sand.	15 to 20 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Filler	Composite from eight to ten sacks, mixing and quartering.	5 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Heated Aggregates	By use of shovel as aggregate flows from storage bin.	5 to 20 lb., depending whether coarse or fine aggregate.	At least daily.	Plant Laboratory.
Asphalt cement A	Standard methods of sampling bituminous materials (A.S.T.M. designation: D 140).	1 qt.	Each car or boat.	Laboratory.
Asphalt cement B	In metal container from valve over asphalt bucket on plant. Mixed and poured into 3-oz. can.	3 oz.	Daily*	Plant Laboratory and Laboratory.
Crude Asphalt	Standard methods of sampling bituminous materials (A.S.T.M. designation: D 140).	1 qt.	Each car or boat.	Laboratory.
Refined Asphalt		1 qt.	Each car or boat.	Laboratory.
Flux		1 qt.	Each car or boat.	Laboratory.
Finished Mixtures — A. To determine average daily analysis of mixture.	Composite from number of batches.	4 oz., minimum, for sheet asphalt; 5 lb., minimum, for asphaltic concrete or binder.	Daily.	Laboratory.
Finished Mixtures — B. For determination of uniformity of individual or different batches.	From individual batches.	4 oz., minimum, for sheet asphalt; 5 lb., minimum, for asphaltic concrete or binder.	As directed.	Laboratory.
Asphalt Block	Select blocks representative of production.	2 blocks.	Daily.	Laboratory.

*When fluxing at paving plants, preliminary 3-oz. samples shall also be taken from each melting kettle, when ready and completely mixed. This is in addition to daily 3-oz. sample as provided above.

(D) SAMPLING BITUMINOUS LACQUERS, CEMENTS, VARNISHES AND JAPANS; ALSO BITUMINOUS EMULSIONS

These classes of products shall be sampled as described for "Crude, Refined and Blended Bituminous Substances," "Sampling at Point of Delivery," under Section (*b*), "Liquid Materials."

CHAPTER XXXII

EXAMINATION OF CRUDE, REFINED AND BLENDED BITUMINOUS SUBSTANCES

The present chapter will be devoted to a description of the most important tests used for examining crude, refined and blended bituminous substances. Certain of the tests have been adopted as standards by technical societies, whose committees have been active in this field, accomplishing much to clarify what formerly constituted a veritable jumble of rule-of-thumb methods. Other tests appearing in the current literature will be included where they have been found adequate,[1] but in certain cases these have been amplified or elaborated to conform with the practice followed in the author's laboratory.

The tests which follow are grouped under four headings, viz., physical characteristics, mechanical tests, thermal tests, solubility tests and chemical tests, a section being devoted to each. In general, a test may have one or more objects in view, viz.:

(1) Serving as a means of identification.[2]
(2) Ascertaining the value of the substance for a given use.
(3) Gauging the uniformity of its supply.
(4) An aid to factory control in its manufacture, refining or blending, and
(5) As a criterion of its quality.

The last named may serve as an indication of its purity, the care exercised in its preparation, or its intrinsic value. The tests pertaining to bituminous substances fulfil these requirements as noted in Table LXXXIII.

Table LXXXIV contains a list of the principal bituminous substances, together with such physical and chemical characteristics as will enable them to be distinguished one from another. Under each heading the minimum and maximum figures are included, based on the author's experience. His intention has been to make the range

TABLE LXXXIII

TEST REQUIREMENTS.

Number	Description	For Purposes of Identification	Adaptability for a Given Purpose	Gauging the Uniformity of Supply	Purposes of Factory Control	As a Criterion of the Quality*
	(A) Physical Characteristics					
(Test 1)	Color	YES				
(Test 2)	Homogeneity	YES	Yes	Yes	Yes	YES
(Test 3)	Appearance surface aged one week	Yes	YES			YES
(Test 4)	Fracture	YES				
(Test 5)	Lustre	Yes	YES			YES
(Test 6)	Streak	YES	Yes			
(Test 6*a*)	Water absorption		YES			Yes
(Test 6*b*)	Diffusibility		YES			Yes
(Test 7)	Specific gravity	YES		Yes	Yes	
(Test 7*f*)	Voids		Yes	Yes	YES	YES
(Test 7*g*)	Colloidal capacity	YES	YES	YES	Yes	YES
	(B) Mechanical Tests					
(Test 8)	Viscosity		YES	Yes	Yes	
(Test 9)	Hardness	YES	YES	YES	YES	
(Test 9*d*)	Susceptibility index	YES	YES	YES	YES	
(Test 10)	Ductility		YES	Yes	Yes	
(Test 11)	Tensile strength (cohesiveness)		YES			
(Test 12)	Adhesiveness		YES			
(Test 12*d*)	Surface tension	YES	Yes			Yes
	(C) Thermal Test-					
(Test 12*f*)	Thermal conductivity	YES	YES			
(Test 12*g*)	Specific heat	YES	YES			
(Test 12*h*)	Heat content	YES	YES			
(Test 12*i*)	Thermal expansion	YES	YES			
(Test 13)	Breaking-point	YES	YES	YES	YES	
(Test 14)	Solidifying-point	YES	YES	YES	YES	
(Test 15)	Softening-point	YES	YES	YES	YES	
(Test 15*g*)	Flow-point	YES	YES	YES	YES	
(Test 15*h*)	Liquefying-point	YES	YES	YES	YES	
(Test 15*i*)	Twisting-point	YES	YES	YES	YES	
(Test 16)	Volatile matter	Yes	YES	YES	YES	Yes
(Test 16*a*)	Evaporation test	Yes	YES	YES	YES	Yes
(Test 16*b*)	Distillation test	Yes	YES	YES	YES	Yes
(Test 17)	Flash-point		YES	YES	YES	Yes
(Test 18)	Burning-point		YES			
(Test 19)	Fixed carbon	YES				
	(D) Solubility Tests					
(Test 21)	Soluble in carbon disulfide	YES	YES	YES		YES
(Test 22)	Carbenes	Yes		Yes	Yes	YES
(Test 23)	Soluble in petroleum naphtha	YES	Yes	Yes	Yes	
(Test 24)	Insoluble in benzol ("free carbon")	YES	Yes	Yes		
(Test 24*a*)	Insoluble in other solvents	YES	Yes	Yes		YES
	(E) Chemical Tests					
(Test 25)	Water	Yes				YES
(Test 26)	Carbon	YES				
(Test 27)	Hydrogen	YES				
(Test 28)	Sulfur	YES				
(Test 29)	Nitrogen	YES				
(Test 30)	Oxygen (in non-mineral matter)	YES				
(Test 30*a*)	Molecular weight	YES				
(Test 31)	Tar acids	YES	Yes	Yes		
(Test 32)	Naphthalene	YES				
(Test 33)	Solid paraffins	YES				
(Test 34)	Sulfonation residue	YES				
(Test 35)	Formolite reaction	YES				
(Test 36)	Degree of mercuration	YES				
(Test 37)	Saponifiable constituents	YES		Yes		YES
(Test 38)	Asphaltic constituents	YES				
(Test 39)	Diazo reaction	YES		Yes		YES
(Test 40)	Anthraquinone reaction	YES				
(Test 41)	Liebermann-Storch reaction	YES				

*(*a*) Purity; (*b*) care exercised in its preparation; (*c*) intrinsic value.

TABLE LXXXIV—SYNOPTICAL TABLE OF THE MOST IMPORTANT

Genus	Species	Member	Specific Gravity at 77° F. (of Non-Mineral Matter) (Test 7)	Engler Viscosity at 212° F. (100 ml.) (Test 8a)	Penetration at 77° F. (Test 9b)	Susceptibility Index (Test 9d)	Fusibility °F.* (Tests 15b and 15c)
Bitumens	Petroleums	Non-asphaltic	0.75–0.90	.....	Liquid	.....	<32
		Semi-asphaltic	0.80–0.95	.....	Liquid	.....	<32
		Asphaltic	0.85–1.00	.....	Liquid	.....	<32
	Natural Waxes	Ozokerite	0.85–1.00	.....	5–10	>80	140–200
		Montan wax	0.90–1.00	.....	0–5	>100	170–200
	Natural Asphalts	<10% mineral matter	0.95–1.12	.....	0–350	15–>100	60–325
		>10% mineral matter	0.95–1.15	.....	0–150	30–>100	60–400
	Asphaltites	Gilsonite	1.05–1.10	.....	0–3	>100	250–350
		Glance pitch	1.10–1.15	.....	0–5	>100	250–350
		Grahamite	1.15–1.20	.....	0	>100	350–600
Pyrobitumens	Asphaltic Pyrobitumens	Elaterite	0.90–1.05	.....	Rubbery	.....	Inf.
		Wurtzilite	1.05–1.07	.....	0–5	.....	Inf.
		Albertite	1.07–1.10	.....	0	.....	Inf.
		Impsonite	1.10–1.25	.....	0	.....	Inf.
		Asphaltic pyrobituminous shales	1.50–1.75	.....	0	.....	Inf.
	Non-asphaltic Pyrobitumens	Peat (dry)	0.15–1.05	.....	0	.....	Inf.
		Lignite (dry)	1.00–1.25	.....	0	.....	Inf.
		Bituminous coal	1.20–1.40	.....	0	.....	Inf.
		Anthracite coal	1.30–1.60	.....	0	.....	Inf.
		Lignitic and coal shales	1.30–1.75	.....	0	.....	Inf.
Pyrogenous Distillates	Pyrogenous Waxes	Wax tailings	1.00–1.10	50–100	10–150	20–40	60–100
		Paraffin wax	0.85–0.95	25–50	5–50	>100	100–150
	Petroleum Tars	Carburetted water-gas tar	1.00–1.18	25–50	.....	.....	<0–10
		Oil-gas tar (Low temperature)	0.95–1.10	25–50	.....	.....	<0–20
		Oil-gas tar (High temperature)	1.15–1.35	Over 50	.....	.....	30–100
	Coal Tars	Horizontal gas-retort tar	1.18–1.33	150–650	.....	.....	<0–40
		Inclined gas-retort tar	1.15–1.25	100–300	.....	.....	<0–30
		Vertical gas-retort tar	1.08–1.16	25–50	.....	.....	<0–25
		Coke-oven coal-tar	1.15–1.26	30–100	.....	.....	<0–25
		Blast-furnace coal-tar	0.95–1.10	75–100	.....	.....	<0–25
		Gas-producer coal-tar	1.12–1.20	100–∞	.....	.....	<0–50
		Low-temperature coal-tar	0.95–1.12	25–50	.....	.....	<0–25
	Wood Tars	Pine tar	1.05–1.15	±65	.....	.....	0–50
		Hardwood tar	1.10–1.21	±50	.....	.....	0–20
	Miscellaneous Tars	Peat tar	0.90–1.05	.....	.....	.....	40–60
		Lignite tar	0.85–1.05	.....	.....	.....	60–90
		Shale tar	0.85–0.95	.....	.....	.....	60–90
		Bone tar	0.95–1.05	.....	.....	.....	<0–10
Pyrogenous Residues	Pyrogenous Asphalts	Residual oils	0.85–1.07	.....	100–350	.....	32–110
		Blown petroleum asphalt	0.90–1.07	.....	25–200	8–40	80–400
		Residual asphalt	1.00–1.17	.....	0–150	40–60	80–225
		Sludge asphalt	1.05–1.20	.....	0–150	40–60	80–225
		Wurtzilite asphalt	1.04–1.07	.....	5–20	30–40	150–300
	Petroleum Pitches	Carburetted water-gas-tar pitch	1.10–1.25	.....	0–100	>100	80–300
		Oil-gas-tar pitch	1.15–1.30	.....	0–100	>100	80–300
	Coal-tar Pitches	Horizontal gas-retort-tar pitch	1.25–1.40	.....	0–100	>100	80–212
		Inclined gas-retort-tar pitch	1.25–1.35	.....	0–100	>100	80–212
		Vertical gas-retort-tar pitch	1.15–1.30	.....	0–100	>100	80–300
		Coke-oven-coal-tar pitch	1.20–1.35	.....	0–100	>100	80–300
		Blast-furnace coal-tar pitch	1.20–1.30	.....	0–100	>100	80–212
		Gas-producer-tar pitch	1.20–1.35	.....	0–100	>100	80–212
		Low-temperature-tar pitch	1.10–1.26	.....	0–100	>100	80–200
	Wood-tar Pitches	Pine-tar pitch	1.10–1.20	.....	0–100	>100	100–212
		Hardwood-tar pitch	1.20–1.30	.....	0–100	>100	100–212
	Miscellaneous Pitches	Rosin pitch	1.08–1.15	.....	10–100	>100	120–200
		Peat-tar pitch	1.05–1.15	.....	0–100	>100	100–250
		Lignite-tar pitch	1.05–1.20	.....	0–100	>100	80–250
		Bone-tar pitch	1.10–1.20	.....	0–100	75–100	80–225
		Fatty-acid pitch	0.90–1.10	.....	5–>350	8–40	35–225

*R. and B. Method (Test 15b) for bitumens, asphaltic pyrobitumens, pyrogenous

DISTINGUISHING CHARACTERISTICS OF BITUMINOUS SUBSTANCES

Fixed Carbon, % (Ash-free Basis) (Test 19)	Soluble in Carbon-Disulfide, % (Test 21)	Non-mineral Matter, Insol. in Carbon-disulfide, % (Test 21)	Mineral Ash, % (Test 21)	Carbenes, % (Test 22)	Soluble in Petroleum Naphtha, % (Test 23)	Oxygen in Non-mineral Matter, % (Test 30)	Solid Paraffins, % (Test 33)	Sulfonation Residue, % (in dist. 235–315° C.) (Test 34b)	Tar Acids, % (Test 31)	Saponifiable Matter, % (Test 37e)	Diazo Reaction (Test 39)	Anthraquinone Reaction (Test 40)
½–2	98–100	0–½	0–1	0–½	98–100	0–2	10–25	90–100	0	0–2	No	No
2–5	98–100	0–1	0–1	0–1	95–100	0–3	½–10	85–95	0	0–2	No	No
5–10	98–100	0–1	0–1	0–1	90–100	0–5	0–Tr.	80–95	0	0–5	No	No
½–10	95–100	0–1	0–5	0–3	75–95	0–2	50–90	90–100	0	0–2	No	No
2–10	98–100	0–2	0–2	0–2	80–100	3–6	0–10	0–10	0	50–80	No	No
1–25	60–98	0–40	0–10	0–5	25–95	0–2	0–5	90–100	0	0–2	No	No
5–25	Tr.–90	0–25	10–90	0–5	Tr.–85	0–2	0–5	90–100	0	0–2	No	No
10–20	98–100	0–1	Tr.–1	0–½	40–60	0–2	Tr.	85–95	0	Tr.	No	No
20–30	95–100	0–1	Tr.–5	0–1	20–50	0–2	Tr.	85–95	0	Tr.	No	No
30–55	45–100	0–5	Tr.–50	0–80	Tr.–50	0–2	Tr.	80–95	0	Tr.	No	No
2–5	10–20	70–90	Tr.–10	Tr.–2	5–10	1–5	Tr.	80–90	.....	Tr.–15	No	No
5–25	5–10	80–95	Tr.–10	Tr.–2	Tr.–2	0–2	Tr.	90–98	.....	Tr.	No	No
25–50	2–10	85–98	Tr.–10	Tr.–2	Tr.–2	0–3	Tr.	90–98	.....	Tr.	No	No
50–85	1–6	90–99	Tr.–10	Tr.–2	Tr.–2	0–3	Tr.	90–98	.....	Tr.	No	No
2–25	Tr.–3	15–70	30–85	0–Tr.	0–Tr.	0–3	Tr.–3	90–98	.....	Tr.	No	No
15–35	2–6	15–98	2–80	0–2	0–5	26–44	.....	.....	.....	Tr.–15	No	No
25–50	2–15	65–98	2–25	0–1	5–10	15–28	.....	.....	.....	Tr.–5	No	No
35–75	½–2	75–98	2–25	0–½	0–1	3–18	.....	.....	.....	Tr.–1	No	No
60–90	0–½	75–98	2–25	0	0	1–5	.....	.....	.....	0	No	No
20–45	0–1	15–70	30–85	0	0–½	3–15	.....	.....	.....	Tr.–2	No	No
2–8	98–100	0–2	0–Tr	0–Tr.	95–100	0–2	Tr.–5	95–100	0	Tr.	No	Yes
0–2	99–100	0–½	0–½	0	99–100	0–Tr.	95–100	95–100	0	0	No	No
10–20	95–100	0. 2–5	0–½	0–2	20–75	1–2	0–5	1–25	0	Tr.–2	Yes	Yes
10–25	99–100	0–0 2	0–½	0–2	50–85	1–2	0–5	20–40	0	Tr.	Yes	Yes
15–35	70–90	10–30	0–½	0–2	25–70	1–2	Tr.	Tr.–10	0	Tr.	Yes	Yes
15–40	60–85	16–40	0–½	0–2	20–40	1–3	0–Tr.	0–5	1–4	2–5	Yes	Yes
15–40	80–85	15–20	0–½	0–2	20–60	1–3	0–2	5–10	4–6	2–5	Yes	Yes
15–30	94–98	2–5½	0–½	0–2	60–80	1–3	0–5	5–10	5–11	7½–15	Yes	Yes
14–40	83–97	3–17	0–½	0–2	60–75	1–3	0–Tr.	0–5	1–5	2–5	Yes	Yes
10–25	65–80	10–25	10–15	0–2	50–70	1–3	3–15	15–15	5–15	10–15	Yes	No
10–30	60–95	5–25	0–25	0–2	50–75	1–3	0–3	0–5	3–9	5–15	Yes	Yes
5–15	90–100	0–7	0–1½	0–1	75–90	1–3	3–15	5–25	10–30	15–40	Yes	No
5–15	92–100	0–7½	0–1	0–2	65–95	5–10	0	1–5	10–40	20–60	Yes	No
5–20	95–100	0–5	0–1	0–2	50–90	2–10	0	1–5	5–15	25–85	Yes	No
5–15	97–100	0–3	0–1	0–2	95–100	5–15	5–15	5–15	5–15	5–20	Yes	No
5–20	96–100	0–2	0–1	0–2	98–100	5–10	10–25	10–20	5–20	5–25	Yes	No
5–10	98–100	0–2	0–1	0–2	95–100	1–5	5–15	15–35	0–2	0–5	Yes	No
5–15	95–100	0–5	0–½	0–2	95–100	2–8	0	0–5	5–40	5–50	Yes	No
2–10	98–100	0–½	0–½	0–1	80–99	0–3	0–15	90–100	0	Tr.–5	No	No
5–20	95–100	0–5	0–½	0–10	50–90	2–5	0–10	90–100	0	Tr.–2	No	No
5–40	85–100	0–15	0–1	0–30	25–85	0–2½	0–10	90–100	0	0–2	No	No
5–30	95–100	0–5	0–1	0–15	60–95	3–7	0–½	80–95	0	0–2	No	No
5–25	98–100	0–½	Tr.–2	0–2	50–80	0–2	0–Tr.	90–95	0	Tr.	No	No
25–45	75–98	2–25	0–½	5–10	50–70	0–2	0–5	0–15	0	0–1	Yes	Yes
20–35	70–98	2–30	0–½	5–10	60–80	0–2	0–5	20–40	0	0–1	Yes	Yes
35–65	45–70	30–55	0–½	2–10	10–20	Tr.–2	0	0–3	5–20	Tr.–1	Yes	Yes
30–45	63–78	28–37	0–½	2–10	15–30	Tr.–2	0	2–6	10–15	Tr.–1	Yes	Yes
15–40	70–94	6–30	0–½	2–10	20–40	Tr.–2	0–Tr.	4–7	20–30	Tr.–1	Yes	Yes
17–60	50–92	8–50	0–½	2–10	10–30	Tr.–2	0	0–5	1–12	Tr.–1	Yes	Yes
10–30	45–75	15–35	10–20	1–5	5–25	Tr.–2	2–5	15–25	20–30	Tr.–1	Yes	No
25–45	60–85	15–40	0–2	2–10	10–30	Tr.–2	0–Tr.	0–5	10–15	Tr.–1	Yes	Yes
8–22	85–98	2–15	0–3	1–5	25–60	Tr.–2	2–5	5–20	25–50	Tr.–1	Yes	No
10–25	40–90	2–60	0–1	0–5	25–80	2–8	0	1–4	10–25	45–75	Yes	No
15–35	30–95	5–70	0–1	2–10	15–50	1–5	0	Tr.–5	30–60	60–95	Yes	No
10–20	98–100	0–2	0–1	0–5	90–100	5–10	0	Tr.–5	.....	25–95	Yes	No
10–30	95–100	0–5	0–1	0–5	65–95	2–8	2–5	5–10	25–40	0–5	Yes	No
10–40	95–99	0–2	0–1	0–5	60–85	2–5	1–5	10–25	30–50	0–8	Yes	No
15–25	85–99	1–15	0–½	0–10	75–95	0–2	0	0–5	.....	2–25	Yes	No
5–35	95–100	0–5	0–5	0–5	80–100	2–10	Tr.	0–5	. . .	5–98	No	No

waxes and pyrogenous asphalts; Cube Method (Test 15c) for tars and pitches.

sufficiently liberal to cover all the commercial varieties, and at the same time prevent the range being too broad, since this would result in unnecessary overlapping. Temperatures will be designated either in degrees Fahrenheit or Centigrade.[3]

(A) PHYSICAL CHARACTERISTICS

COLOR

Test 1a. Color in Mass. This test is used largely for purposes of identification, and consists in examining a freshly prepared surface of the bituminous material in daylight. The colors range from white, through the various shades of yellow, brown and black. Some possess a greenish or reddish cast, and again others may appear fluorescent. Purified mineral waxes appear pure white, wax tailings a bright yellow, asphalts and pitches are generally brownish-black, grayish-black or black.

Test 1b. Color in Solution. This test is likewise of value for purposes of identification and consists in observing the color of the bituminous substance when dissolved in a suitable solvent, either in daylight or when viewed in ultra-violet light.[4]

One procedure consists[5] in dissolving 0.25 gram of the substance in 100 ml. of perchlorethylene (CCl_2:CCl_2), filtering, and pouring into a glass cell, the internal distance between the walls being 0.5 ml. The cell is then placed in a Lovibond Tintometer (B.D.H. pattern) and the color of the solution measured by matching it with standard red, yellow and blue glasses. Typical color values are given in Table LXXXV.

For any given type, the harder the asphalt, the darker will be the color. Trinidad asphalt is remarkably light in color for so hard a material. Asphalts recovered from natural rock-asphalts by extraction are all light in color (i.e., they show a comparatively small value of "neutral tint" units in their color). This color test forms a useful guide in ascertaining whether a Trinidad asphalt mixture has been prepared with a flux oil or with a residual petroleum asphalt, also whether the Trinidad asphalt has been fluxed with a natural rock-asphalt.

Another colorimetric test[6] consists in preparing a 0.01 per cent solution of the substance in benzol (freed from mineral constituents), and then titrating an aqueous solution of iodine in potassium

TABLE LXXXV

COLOR VALUES OF ASPHALTS

	Penetration at 77° F. (Test 9b)	Color Value		
		Neutral Tint	Orange	Yellow
Extracted asphalt from:				
St. Jean rock-asphalt	79	1.4	6.3	32.3
Sicilian rock-asphalt	76	2.4	7.1	40.5
Val de Travers rock-asphalt	73	2.0	8.5	49.5
Trinidad asphalt	11	2.1	6.9	4.1
Residual Petroleum Asphalts:				
From Panuco petroleum	45	3 3	11.0	45.7
From Mexican light petroleum	40	7 2	12.8	49.0
From Venezuelan petroleum	42	7 1	12.9	40.0

iodide (1 gram I_2 and 2 grams KI dissolved in 1000 grams distilled water) into pure water, in small cylindrical bottles, until its color in daylight exactly matches that of the former. The number of mls. of the iodine solution which contains 1 mg. of iodine is taken as a measure of the color of the bituminous substance. Representative bituminous substances show a range of from 0.15 to 2.0 ml. iodine solution.

It is claimed that asphalts and tar products when dissolved in solvents may be differentiated by their colors (i.e., fluorescence) when observed in ultra-violet light.[7] Asphalts appear greenish-brown, bitumens greenish-yellow and tar products reddish-brown. Tar-asphalt mixtures may be compared with color standards of known mixtures and their components estimated to within about 5 per cent.

HOMOGENEITY

This test is used for purposes of identification, for determining the adaptability of the bituminous substance to a given purpose, for gauging the uniformity of supply, for purposes of factory control, as a rough criterion of the purity, and when the bituminous mixture is free from mineral and carbonaceous matter, for ascertaining whether a complete amalgamation of the constituents occur, especially after fluxing.

Test 2a. To the Eye at 77° F. With soft materials this may be ascertained by disturbing a freshly prepared surface of the material (cooled to room temperature) with a rod or spatula, and observing whether any dulling occurs. An alternate method consists in drawing a small pellet into a thread between the fingers, and noting whether it dulls while being drawn out. With hard and brittle substances a freshly fractured surface may be examined. Any evidence of dullness is an indication of: (1) the presence of mineral matter, (2) the presence of free carbon (non-mineral matter insoluble in carbon disulfide), (3) an imperfect blending of the bituminous constituents.

Test 2b. Under Microscope. This is ascertained by spreading a minute quantity of the bituminous material on a microscope slide in a thin layer and examining it by transmitted light under a magnification of 100 to 250 diameters. With hard bituminous materials, the slide should be warmed and the specimen spread uniformly and thinly, while melted. This test manifests the same characteristics as the preceding, and in addition, permits the detection of the solid paraffins, which separate from the bituminous matrix in crystal-like masses. Paraffin may be identified positively under a microscope equipped with polariscopic attachment.

Test 2c. When Melted. This constitutes a rough test for detecting the presence of substantial amounts of extraneous matter, such as mineral constituents or free carbon. The bituminous material is simply melted and stirred with a rod. If these constituents are present in large quantities, they will impart a gritty feel to the mass, and will often settle out on standing.

Test 2d. In Solution.[8] A weight of the sample equivalent to 2 ml. is placed in a 25-ml. Erlenmeyer flask, and if it does not flow readily at room temperature, it is cautiously heated over a hot-plate until it flows over the bottom. Upon cooling, add 10.2 ml. of straight-run petroleum naphtha, free from cracked products of any kind, conforming to the following requirements: A.P.I. gravity 49 to 50; initial boiling-point above 300° F.; 50 per cent distils over at 335 to 355° F.; end-point below 410° F.; aniline number (A.S.T.M. D 91–33) 138 to 145° F. Insert a cork stopper in which is fitted an 8-in. length of open ¼-in. glass tubing, and swirl the contents of the flask with a rapid circular motion for 5 sec.,

whereupon the flask is immediately immersed to its neck in a bath of gently boiling water. The contents of the flask shall again be swirled for 5 sec. at the end of each minute thereafter until complete dispersion takes place. If the dispersion is not completed in from 6 to 8 min., the test shall be repeated with the bath maintained at a temperature 25° F. lower or higher (glycerin being substituted for the water in the latter case) until dispersion is effected within the stipulated time. After complete dispersion, the flask is allowed to cool to room temperature, and if any loss in weight occurs, it is made up with additional solvent. A drop of the asphalt-solvent mixture is then placed on a No. 50 Whatman filter-paper. If the drop forms a brown or yellowish-brown circular stain, with a darker, solid or annular nucleus in the center, the product is classified as "heterogeneous." If, however, the drop forms a uniformly brown circular stain, judgment shall be reserved, and the solution shall be set aside in its tightly-stoppered flask at room temperature in a subdued light, for a period of 24 hr. The mixture shall then be vigorously agitated till uniform, and a drop shall again be placed on the filter-paper. If the drop from the 24-hr. old mixture still forms a uniformly brown circular stain, the product is classified as "homogeneous"; but if a darker, solid or annular nucleus, as described above now forms in the center of the stain, the product shall be classified as "hetrogeneous."

A quantitative method, based on the foregoing, consists in running the spot test as described, but instead of employing 10.2 ml. of straight naphtha, exactly the same volume of a predetermined mixture of xylene and standard petroleum naphtha is employed. If the resulting solution, which should be completed within 6 to 8 minutes, tests positive within 24 hr., a fresh solution is prepared, but with a larger proportion of xylene in the mixed solvent, the volume of xylene being progressively increased in increments of 1.0, 0.5, or 0.1 ml. at a time, until a negative test is obtained at the end of 24 hr. The minimum percentage of xylene by volume in the mixed solvent necessary to assure a negative spot for a given asphalt, is reported as the "xylene equivalent" of that asphalt.

This test is stated to identify all petroleum asphalts that have been subjected to temperatures higher than are normal in the regular steam-refining processes, including cracked asphalts, steam-re-

fined or vacuum-process residuals that have been subjected to higher temperatures than are normal, the more highly blown asphalts that have been subjected to a higher temperature for prolonged periods of time, the presence of waxy bodies, acid-sludge bodies, the combination of incompatible fractions in so-called "synthetic" asphalts, and exposure to the air or weathering.

APPEARANCE SURFACE WHEN AGED

Test 3. Appearance Surface Aged Indoors One Week. A small quantity of the bituminous material is carefully melted at the lowest possible temperature and poured into a tin ointment box or deep seamless can as used for determining the volatile matter (Test 16). The surface should be free from froth or bubbles and allowed to cool in a place free from draughts. When cool, the surface is examined, and then covered to protect it from dust. At the end of a week the cover is removed and the surface re-examined. If bright and lustrous, it will indicate a perfect amalgamation of the constituents, also the absence of oily, greasy and undissolved constituents. A lustreless surface is an indication of the presence of extraneous mineral or carbonaceous matter, or evidence that the constituents do not blend or amalgamate properly. If the surface appears greasy or wax-like, vaseline- or paraffin-like bodies are present, since these have the property of separating or "sweating" from the bituminous matrix on standing. This would prove objectionable where the bituminous material is to be used for surfacing prepared roofings dusted with talc, or for manufacturing bituminous paints, varnishes or japans. This test is accordingly used for purposes of identification, determining the adaptability of the substance for a given purpose and as a criterion of its quality.

FRACTURE

Test 4. Conventional Method. This is ascertained upon cleaving the specimen by subjecting it to a sharp blow, and examining the cleavage surface. Only hard and "brittle" bituminous substances will yield to this test, including the hard asphalts and asphaltites. The fracture may either appear conchoidal (rounded and curved like a shell), or hackly (jagged, irregularly and rough),

LUSTRE

Test 5. Conventional Method. This indicates the manner in which light is reflected from a freshly fractured surface, and may be bright or vitreous—indicating that it has the brilliancy or shine of glass; greasy—indicating that it presents an oily or greasy surface; waxy—indicating that it has the characteristic appearance of wax; or dull—indicating that the surface is without lustre. These manifestations are used for purposes of identification, and for determining the adaptability of the bituminous material for manufacturing paints, varnishes and japans.

STREAK

Test 6. Conventional Method. This represents the color of the powder which is left behind on drawing a piece of the solid bituminous material across the surface of unglazed porcelain. Hard bituminous materials only will yield to this test. The streak may be classified as white (where no streak is visible), yellowish, yellowish-brown, brown, brownish-black and black. This test is of value for purposes of identification, and as an indication of the suitability of the substance for use with colored pigments.

WATER ABSORPTION

Test 6a. Quantitative Method. The capacity of bituminous substances to absorb water may be ascertained by the following method: clean brass plates measuring 3 by 4 by $\frac{1}{32}$ in. (of which the surface area measures 21 sq. in., excluding the edges) are heated to 250° F. and dipped into the melted asphalt to a depth of $3\frac{1}{2}$ in., so as to give a film weighing 7.5–8.0 grams in one dip. Each plate is then heated carefully over a burner to expel the air from the substance and to seal the edges. Weigh and immerse in distilled water at room temperature for 52 weeks. Remove the plates, dry the surface quickly with a sheet of filter paper and reweigh. In this test blown asphalts derived from petroleum showed increases in weight ranging from 1.5 per cent to 12.0 per cent (Colombian petroleum asphalt the lowest and mid-continental asphalt the highest). At the end of 156 weeks, the increases in weight ranged from 2.8 per cent to 21.3 per cent respectively, as illustrated in Fig. 199A.

FIG. 199A. WATER-ABSORPTION TEST ON BLOWN PETROLEUM ASPHALTS.

		(1) Blown Mid-Continental Asphalt	(2) Blown Mexican Asphalt	(3) Blown Venezuelan Asphalt	(4) Blown West-Texas Asphalt	(5) Blown Colombian Asphalt
(Test 7)	Specific gravity at 77° F. . .	1 006	1.055	1 037	1 026	1.099
(Test 9*b*)	Penetration at 115° F. . . .	26	17½	20	16	26
	Penetration at 77° F . ..	17	10	13	9½	17
	Penetration at 32° F	8½	6	7	5½	10
(Test 15*b*)	Fusing-point (R. & B.).	230° F.	242° F.	234° F.	221° F.	230° F.
(Test 19)	Fixed carbon.	16 36%	19.48%	19 00%	17.15%	15 95%
(Test 38*c*)	Asphaltenes.	33 6%	46 2%	40 2%	38 4%	36 2%
(Test 38*d*)	Asphaltic resins	25 5%	16 0%	17.1%	20 9%	15 6%
(Test 38*e*)	Oily constituents.	39 1%	37 0%	41.7%	40 0%	47.1%

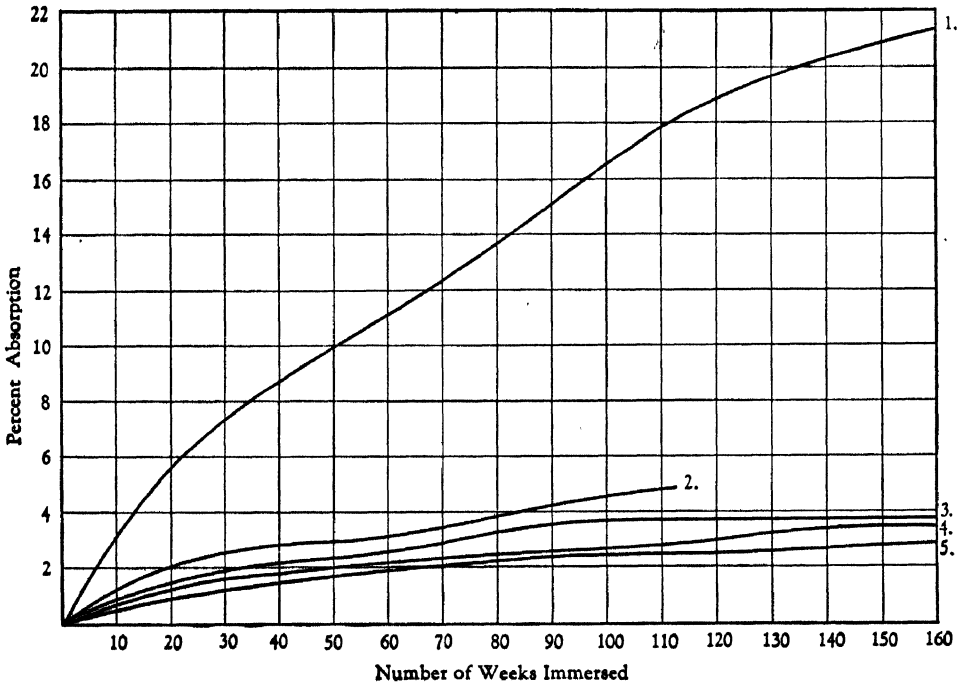
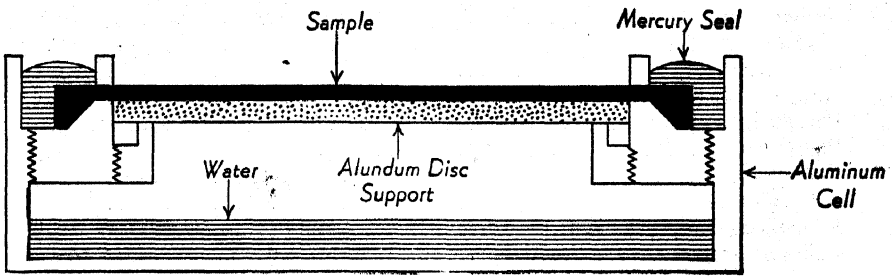


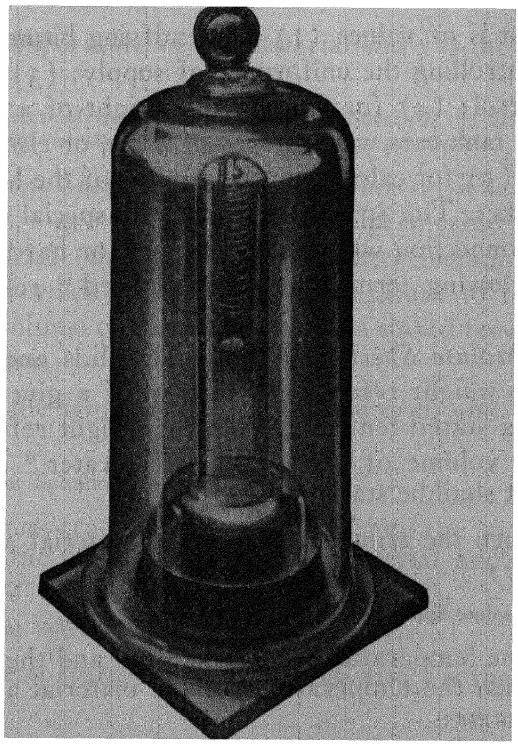
FIG. 199A.

DIFFUSIBILITY

Test 6b. Quantitative Method. The "diffusibility constant" is taken as the number of grams of water vapor which will diffuse through a 1 cm. cube of the substance in 1 hour under a vapor pressure difference of 1 mm. mercury at 77° F.[9] The bituminous



A



B

FIG. 200.—Apparatus for Ascertaining Diffusibility. A—Cross-section of Cell; B—Apparatus Assembled.

substance is mounted on an aluminium cell of the form illustrated in Fig. 200. The cell is placed in a desiccator maintained at 77° F., with a drying agent attached to a calibrated spring, which serves to give direct readings of the weight of water absorbed. The following figures show the diffusibility $\times 10^8$:

Hydrocarbon wax	0.1
Thiokol	0.75
Asphalt	1.25
Gutta percha	1.5
Hard rubber (ebonite)	1.5
Paragutta	2.
Polystyrene	3.5
Phenol fiber	6.5
Soft vulcanized rubber	7.
Benzyl cellulose	10.5
Cellulose acetate	160.

SPECIFIC GRAVITY

This test is of value: (1) in identifying bituminous materials; (2) for controlling the uniformity of supply; (3) for purposes of factory control; (4) for figuring the weight of a given volume as delivered in tank cars, when stored in tanks, or else upon filling into containers; (5) for calculating the volume of the bituminous binder in pavements. The specific gravity is of special value when considered in connection with the fusing-point or hardness.

The following terms have been defined [10] relating to specific gravity:

(*A*) *Absolute Specific Gravity* (of solids and liquids). The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water * at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same*:

Absolute Specific Gravity $x°/x°$ C. .....,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same*:

Absolute Specific Gravity $x°/y°$ C. .....,

* Distilled water boiled vigorously in vacuum.

where x is the temperature of the material and y the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

(*B*) *Specific Gravity* (of solids and liquids). The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same*:

Specific Gravity $x°/x°$ C. .....,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same*:

Specific Gravity $x°/y°$ C. .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

(*C*) *Apparent Specific Gravity* (of solids). The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same*:

Apparent Specific Gravity $x°/x°$ C. .....,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same*:

Apparent Specific Gravity $x°/y°$ C. .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by definition B would be designated

as "apparent specific gravity" and that defined by definition A as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

NOTE 2.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

NOTE 3.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

(*D*) *Bulk Specific Gravity* (of solids). The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same*:

Bulk Specific Gravity $x°/x°$ C. .....,

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same*:

Bulk Specific Gravity $x°/y°$ C. .....,

where x is the temperature of the material and y is the temperature of the water.

NOTE 1. See Note 2 under "Apparent Specific Gravity."

NOTE 2. In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

Density, i.e., mass per unit volume, is a much more fundamental concept than specific gravity, and there is an increasing tendency to employ density instead of specific gravity, both in scientific and in industrial work.[11] Densities are generally most conveniently expressed in terms of grams per milliliter. The density of water at 15.5° C. is 0.999050 g./ml., so that, if S (15.5° C./15.5° C.) is the specific gravity of a liquid as defined above, the density at 15.5° C. is S (15.5° C./15.5° C.) × 0.999050 g./ml. The numerical difference between a specific gravity S (15.5° C./15.5° C.) and the corresponding density in grams per milliliter at 15.5° C. is therefore approximately one part in a thousand. Since the liter is defined as the volume of one kilogram of water at its temperature of maximum density (i.e., 4° C.), it follows that the specific gravity

S ($t°$ C./4° C.) of a substance is numerically identical with its density at the temperature $t°$ C. expressed in grams per milliliter.

Table LXXXVI gives the corrections, expressed as units in the fourth decimal place, to be subtracted from the specific gravity S ($t°$ C./$t°$ C.) in order to obtain the corresponding density in grams per milliliter at $t°$ C.:

TABLE LXXXVI

CONVERSION OF SPECIFIC GRAVITY TO DENSITY

Sp. Gr.	$S_{0° C/0° C}$	$S_{15.5° C/15.5° C}$	$S_{25° C/25° C}$	$S_{38° C/38° C}$
0.6	1	6	18	42
0.7	1	7	20	49
0.8	1	8	23	56
0.9	1	9	26	63
1.0	1	10	29	70
1.1	1	10	32	77
1.2	2	11	35	84
1.3	2	12	38	91

Example.—The density at 25° C. in g/ml of a liquid having a specific gravity $S_{25° C/25° C}$ of 0.8564 is obtained as follows:

The correction given in the table for $S_{25° C/25° C}$ of 0.8 is 23, and for $S_{25° C/25° C}$ of 0.9 is 26, and therefore, by interpolation, the correction for $S_{25° C/25° C}$ of 0.8564 is 25. Hence, the density at 25° C. of a liquid having a specific gravity $S_{25° C/25° C}$ of 0.8564 is (0.8564 − 0.0025) g/ml, i.e. 0.8539 g/ml.

When specific gravity is required with accuracy in the fourth place of decimals, and is determined by a weighing method (bottle, pyknometer, etc.) it is necessary to apply corrections for the buoyancy effect of the air as follows:

If W_w be the observed weight in air of the water content at 15.5° C., and W_s be the observed weight in air of the sample contained in the bottle (or pyknometer, etc.) at 15.5° C.; then the *approximate* specific gravity $S^1_{15.5° C/15.5° C}$ is given by the equation:

$$S^1_{15.5° C/15.5° C} = \frac{W_s}{W_w} \qquad (1)$$

The value given by the above formula may be corrected for the buoyancy effect of the air by means of the following equation:

$$S^1_{15.5° C/15.5° C} = S^1_{15.5° C/15.5° C} - 0.0012\,(S^1_{15.5° C/15.5° C} - 1) \qquad (2)$$

where $S^1_{15.5^\circ C/15.5^\circ C}$ is the value obtained from (1) and $S_{15.5^\circ C/15.5^\circ C}$ is the corrected specific gravity.

Table LXXXVII gives the values of $-\{0.0012\,(S^1_{15.5^\circ C/15.5^\circ C}-1)\}$, expressed as units in the fourth decimal place, to be added to (when the sign is positive) or subtracted from (when negative) the approximate specific gravity $S^1_{15.5^\circ C/15.5^\circ C}$, to give the corrected specific gravity $S_{15.5^\circ C/15.5^\circ C}$.

TABLE LXXXVII

BUOYANCY CORRECTIONS—VALUES OF $-\{0.0012\,(S^1_{15.5^\circ C/15.5^\circ C}-1)\}$ AS UNITS IN FOURTH DECIMAL PLACE

$S^1_{15.5^\circ C/15.5^\circ C}$	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
0.60	+5	+5	+5	+4	+4	+4	+4	+4	+4	+4
0.70	+4	+3	+3	+3	+3	+3	+3	+3	+3	+3
0.80	+2	+2	+2	+2	+2	+2	+2	+2	+1	+1
0.90	+1	+1	+1	+1	+1	+1	0	0	0	0
1.00	0	0	0	0	−1	−1	−1	−1	−1	−1
1.10	−1	−1	−1	−2	−2	−2	−2	−2	−2	−2
1.20	−2	−3	−3	−3	−3	−3	−3	−3	−3	−3
1.30	−4									

In the United States, it is customary to ascertain the specific gravity of bituminous materials at 77° F./77° F., petroleum products at 60° F./60° F., and creosote oils at 100° F./60° F.

For converting the specific gravity of a substance found at a higher temperature to the standard temperature (lower), the following formula should be used:

Sp. gr. Substance at t_1/t_1 = Sp. gr. Substance at $t_2/t_1 + k(t_2 - t_1)$, in which

t_2 = the temperature at which the specific gravity of the substance was determined,

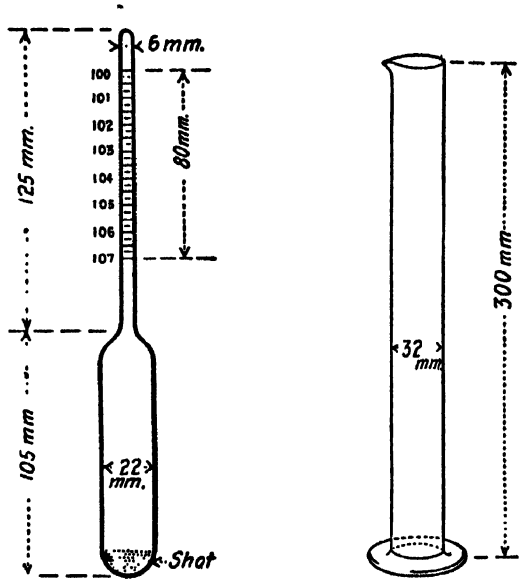
t_1 = the temperature (lower) at which the specific gravity of the substance is to be calculated, and

k = the coefficient of cubical expansion per deg. F., which is constant for the particular substance.

If perchance the specific gravity of the substance has been compared with that of water at a higher temperature, then to convert it to a lower temperature compared with water at the same temperature, the following formula should be used:[12]

Sp. gr. Substance at t_1/t_1 = Sp. gr. Substance at $t_2/t_2 \times$ Sp. gr. Water at $t_2/t_1 + k(t_2 - t_1)$.

Test 7a. Hydrometer Method.[18] Where speed is essential and great accuracy not required, the specific gravity of fluid bituminous materials may be determined with a hydrometer having its scale sub-divided to unity in the third place of decimals. Usually a series of hydrometers are used, ranging respectively from 0.800 to 0.900, 0.900 to 1.000, 1.000 to 1.080, 1.070 to 1.150, 1.150 to 1.230, and in such dimensions as to enable them to be used in a 100 ml.



Courtesy A.S.T.M.

FIG. 201.—Hydrometer for Liquid Bituminous Substances.

cylinder approximately 300 mm. long having an inside diameter of not less than 32 mm. as illustrated in Fig. 201. The hydrometer shall conform to the following requirements as to dimensions:

	Dimension	Permissible Variation
Length of stem	125 mm.	6 mm.
Length of bulb	105 mm.	5 mm.
Length of scale	80 mm.	4 mm.
Diameter of stem	6 mm.	0.5 mm.
Diameter of bulb	22 mm.	1 mm.

Most hydrometers are adapted to read at 60° F./60° F., or in other words, the instruments are calibrated for water at 60° F. taken as unity. The standard temperature for testing bituminous materials is 77° F., and they should accordingly be brought to this

temperature when tested with the hydrometer. For correcting the reading to water at 77° F., it should be multiplied by 1.002, as follows:

Sp. gr. at 77° F./77° F. = Sp. gr. at 77° F./60° F. × 1.002.

In running the test, the bituminous material is brought to 77° F., immediately poured into the hydrometer jar, and then the hydrometer allowed to sink into it until it comes to a definite resting-point, whereupon it is raised slightly, and allowed to sink a second time. The reading is then noted. The hydrometer must not be pushed below the point at which it comes to rest until after the second reading has been taken, whereupon it should be pushed a slight distance below the end-point to observe whether or not it will rise. If it fails to do so, it is evident that the bituminous material is too viscous to be tested by the hydrometer method, and some other method should be employed. Care should be taken that the hydrometer does not touch the sides or bottom of the cylinder when the reading is taken, also that the surface of the liquid is free from froth or bubbles.[14]

The hydrometer has also been recommended for ascertaining the specific gravity of hard asphalts,[15] which are melted and tested while fluid at elevated temperatures, the results being converted to 60° F./60° F. by suitable conversion tables.

For converting specific gravity into degrees Baumé and vice versa, the following formulae may be used:[16]

For liquids lighter than water:

$$°\text{Baumé} = \frac{140}{\text{Sp.gr. } 60° \text{ F.}/60° \text{ F.}} - 130$$

$$\text{Sp.gr. } 60° \text{ F.}/60° \text{ F.} = \frac{140}{130 + °\text{Baumé}}$$

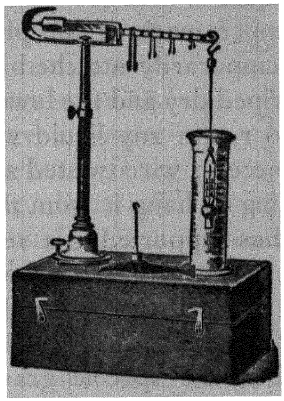
For liquids heavier than water:

$$°\text{Baumé} = 145 - \frac{145}{\text{Sp.gr. } 60° \text{ F.}/60° \text{ F.}}$$

$$\text{Sp.gr. } 60° \text{ F.}/60° \text{ F.} = \frac{145}{145 - °\text{Baumé}}$$

Crude petroleum and refined liquid petroleum products are generally reported in terms of "A.P.I. Gravity" [17] as follows:

$$°\text{A.P.I.} = \frac{145.1}{\text{Sp.gr. } 60° \text{ F.}/60° \text{ F.}} - 131.5$$



Courtesy of Eimer & Amend.

FIG. 202.—Westphal Balance.

Test 7b. Westphal Balance Method. This is also adapted to testing fluid bituminous materials. The instrument as supplied by the manufacturer (Fig. 202) is provided with a cylinder of about 50 ml. capacity, calibrated for use at 60° F./60° F. Since the test is generally made at 77° F., it is subject to the same correction as in the hydrometer method.

The Westphal balance may be adapted for as little as 8 ml. of the bituminous material, by using a special plummet, small enough to fit into a 10-ml. cylinder. The plummet may be made from a piece of glass tubing 7 mm. outside diameter, which is sealed at one end with a short platinum wire fused into the glass. Nine to ten grams of mercury are placed in the tube, forming a column 35–40 mm. high. The tube is then cut off within 20 mm. of the top of the mercury column, and the open end sealed with a blowpipe. This plummet should measure 55–60 mm. over all, and weigh from 10 to 12 g. If a represents the weight required to balance the plummet in air, b the weight required to balance the plummet in water at a definite temperature, and c the weight required to balance the plummet in the bituminous material at the same temperature, then the specific gravity of the bituminous material at this temperature $= \frac{c - a}{b - a}$.

Test 7c. Bottle Method. Several forms of glass bottles are used for this purpose, having a ground-glass stopper with a small vertical hole bored through, to enable it to be completely filled with the bituminous material. These are made in various sizes.

An improvised form, which may be used to good advantage when a small quantity of liquid bituminous material is available, consists of a 1-ml. pipette, and a glass tube sealed at one end, the

inside diameter of which is slightly larger than the outside diameter of the lower stem of the pipette. On using this instrument, the liquid is first brought to a definite temperature, then sucked to the upper mark of the pipette by means of a piece of rubber tubing temporarily attached to its upper stem. The outside is carefully wiped dry and the lower stem inserted in the glass tube which serves to retain any liquid which may drain from the pipette. A small piece of wire twisted about the pipette near the top is formed into a ring to hang it from the hook above a balance pan. The pipette is thus supported in a vertical position and weighed.[18]

If a represents the weight of the pipette with glass tube empty, b its weight filled with water at a definite temperature, and c its weight filled with the bituminous material at the same temperature, then the specific gravity may be calculated from the following formula:

$$\frac{c - a}{b - a}$$

If only a small quantity of the substance is available, the following procedure is recommended:[19] the specific-gravity bottle is weighed empty (a), a small piece of the substance is pressed against the interior wall of the bottle and again weighed (b). The bottle is thereupon filled with water at 77° F. and again weighed (c). If (d) is the weight of the bottle filled with water at 77° F. alone, then the specific gravity at 77° F. of the substance may be calculated as follows:

$$\frac{b - a}{d + b - (a + c)}$$

Test 7d. Pyknometer Method.[20] This method has been standardized as follows:[21]

(A) The specific gravity of road oils, road tars, asphalt cements and soft tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific Gravity 25° C. (77° F.)/25° C. (77° F.)

The determination of specific gravity shall be made with a pyknometer or weighing bottle (Fig. 203), which shall consist of a straight-walled glass tube approximately 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.5 to 1.7-mm. bore in place of the usual capillary opening. The lower part of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression shall be about 4.8 mm. at the center. The stoppered tube should have a capacity of about 24 ml. and when empty should weigh not over 35 g.

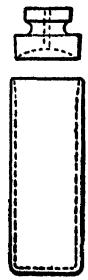


FIG. 203.—Pyknometer or Weighing Bottle.

Before making a determination, the pyknometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called *a*. It shall then be filled with freshly boiled distilled water at a temperature of 25° C. (77° F.), the stopper firmly inserted, all surplus moisture wiped from the surface with a clean dry cloth and again weighed. This weight is called *b*.

When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25° C. (77° F.) and poured into the pyknometer until it is full, with care to prevent the inclusion of air bubbles. The stopper is then firmly inserted and all excess of material forced through the opening is carefully removed with a clean dry cloth. The pyknometer and contents are then weighed and this weight is called *c*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c - a}{b - a}$$

When determining the specific gravity of tar and asphalt products which are too viscous for the method described in the preceding paragraph, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss by evaporation. When sufficiently fluid, enough is poured into the clean dry pyknometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air

bubbles. The tube should be slightly warmed before filling. The pyknometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called *c*. The pyknometer is next removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for not less than thirty minutes in a beaker of distilled water maintained at 25° C. (77° F.) after which it is removed, and all surplus water is wiped off with a clean cloth. It is immediately weighed. This weight is called *d*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c - a}{(b - a) - (d - c)}$$

When making the specific gravity determination it is important that:

(*a*) Only freshly boiled distilled water shall be used.

(*b*) When weighing the pyknometer completely filled, the temperature of its contents shall be within 1° C. (1.8° F.) of 25° C. (77° F.).

(*c*) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pyknometer.

(*d*) The presence of all air bubbles shall be eliminated in filling the pyknometer and inserting the stopper.

(*e*) Weighings shall be made quickly after filling the pyknometer and shall be accurate to 1 mg. A number of trial fillings and catch weights may be necessary to obtain the desired degree of accuracy.

(*f*) To prevent breakage of the pyknometer when cleaning it out after a determination has been made upon a very viscous or semi-solid material, it will be found advisable to warm it in an oven at not over 100° C. until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool it may be finally rinsed with carbon disulfide, benzol or other solvent and wiped clean.

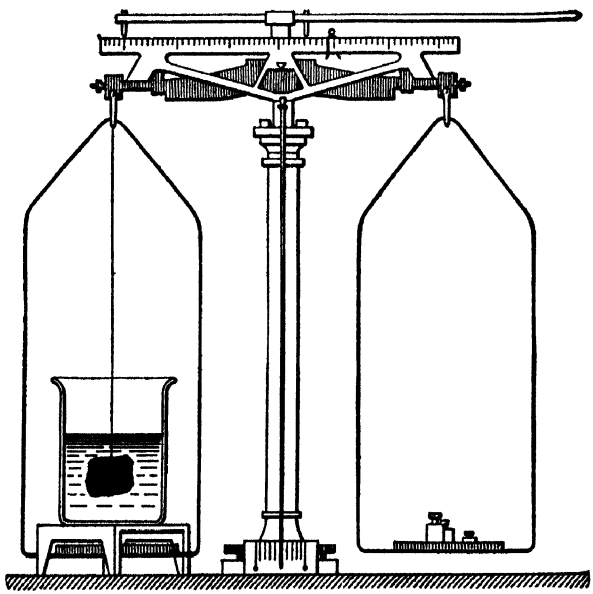
The limit of accuracy of the test is ± 0.005 specific gravity.

(*B*) The specific gravity of creosote oil fractions are determined in a similar manner by means of a pyknometer, but in this case the results are expressed at 38° C. (100° F.)/15.5° C. (60° F.).

(*C*) The pyknometer method may also be used for finding the specific gravity of hard and brittle bituminous substances,[22] includ-

ing hard asphalts of high fusing-point, asphaltites, asphaltic pyrobitumens, non-asphaltic pyrobitumens and pyrobituminous shales. Approximately 3.5 grams of the material ground to 60-mesh are carefully weighed and introduced into a 50-ml. pyknometer, with about 30 ml. of distilled water. A vertical condensing bulb is attached to the pyknometer with a small section of rubber tubing, the open end being connected with an aspirator to maintain a partial vacuum. The pyknometer is then boiled on a water-bath to expel all the air from the sample. The inside of the condensing tube is then washed back into the pyknometer, which is cooled to the desired temperature, stoppered, filled to the mark with water at the same temperature and weighed. The specific gravity may then be calculated from the foregoing formula.

Test 7e. Analytical Balance Method. This method is adapted for substances which can be melted and cast in a mold to form a



Courtesy A.S.T.M.

FIG. 204.—Analytical Balance for Ascertaining the Specific Gravity.

briquette which may be handled on cooling. It has been standardized as follows: [23]

The determination of specific gravity shall be made with an analytical balance equipped with a pan-straddle or other stationary support (Fig. 204). The test specimen shall be a cube of the material measuring approximately ½ in. to the edge. It shall be

prepared by melting a small sample of the material by the gentle application of heat, care being exercised to prevent loss by evaporation, and pouring when sufficiently fluid into a ½-in. brass cubical mold, having the form illustrated in Fig. 254, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. Precautions should be taken to prevent the inclusion of air bubbles. The hot material should slightly more than fill the mold and when cool the excess may be cut off with a hot spatula. The specimen shall be removed from the mold when cooled to room temperature.

The balance shall first be tared with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. The test specimen shall then be attached to the thread, so as to be suspended about 1 in. above the straddle from the hook on the pan support, and weighed. This weight is called *a* and shall be accurate to 0.1 mg. The specimen, still suspended by the thread, shall then be weighed completely immersed in freshly boiled distilled water at 25° C. (77° F.) ± 1° C. (1.8° F.), adhering air bubbles being first removed with a fine wire. This weight is called *b* and shall be accurate to 0.1 mg. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{a}{a - b}$$

The limit of accuracy of the test is ± 0.005 specific gravity.

VOIDS

Test 7f. Voids (Entrapped Air). The voids in asphalts, asphalt-filler mixtures and bituminized fabrics may be rapidly determined as follows: The volume of the specimen is first ascertained from its specific gravity, or in the case of bituminized fabrics, from its linear dimensions and thickness in cms. The sample weighing 25 to 50 g. is then placed in the reservoir "A" of a graduated funnel, as shown in Fig. 205. A copper wire-gauze is inserted in the bottom to support the sample, whereupon the funnel is inverted, and with the stop-cock open, immersed in a cylinder filled with carbon tetrachloride. When the funnel is full and the carbon tetrachloride passes through the stop-cock, the latter is quickly closed. The cylinder containing the funnel is placed in a water-bath maintained at 50° C. The entrapped air rises to the top and displaces the carbon tetrachloride. After the bubbling has ceased, the cylin-

der and contents are cooled to 20° C., and the volume of air is then read. This divided by the volume of the sample gives the volume percentage of the voids. When tested in this manner, re-

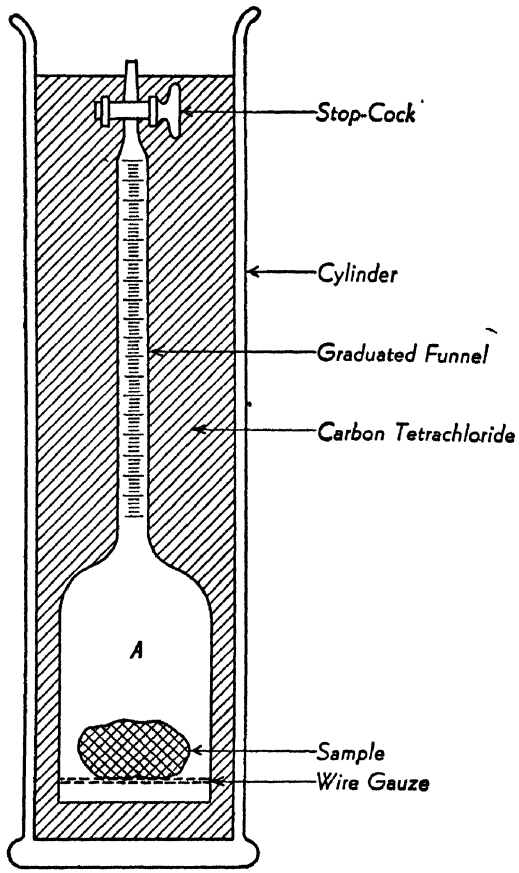


FIG. 205.—Apparatus for Ascertaining Voids (Entrapped Air).

sidual asphalt showed 2 per cent of air; filled asphalt 6 per cent; asphalt roll-roofings 2 to 18 per cent by volume of entrapped air (i.e., voids).

COLLOIDAL CAPACITY

The tests included under this heading are designed for the purpose of measuring the capacity of bituminous substances to maintain colloidal dispersions of finely divided inorganic materials. This depends upon the characteristics of the bituminous substance, as well as the nature and physical subdivision of the inorganic material present. Colloidal dispersions of this type are characterized by

the fact that the inorganic material remains in more or less permanent suspension upon dissolving the bituminous substance in carbon disulfide or benzol. The test is of value in measuring the stability of bituminous substances combined with finely divided inorganic materials and the ability of the latter to remain in dispersion. The following tests have been proposed for the purpose:

Test 7g-1. Clay Dispersions. This test has been devised by Clifford Richardson,[24] and measures the capacity of the bituminous substance to maintain a dispersion of colloidal clay. A suitable type of colloidal clay is first selected and dispersed in water. This dispersion is then introduced into the bituminous substance maintained in a liquid condition below the temperature of boiling water. Upon removing the aqueous phase from the emulsion by heating, the highly dispersed clay will be associated with the bituminous phase, and become dispersed in a colloidal state to an extent depending upon the colloidal capacity of the bituminous substance. Tests were made by the foregoing method to introduce colloidal clay into a number of solid residual asphalts derived from various types of petroleum, which resulted in a product of approximately 33⅓ per cent clay combined with 66⅔ per cent asphalt, after removal of the water. The following results were recorded:

	Original Penetration at 77° F. (Test 9*b*)	Quantity of Colloidal Clay in Dispersion	Mixture Maintained in Tubes 24 Hrs. at 325°F		Clay Remaining in Suspension Upon Centrifuging a 10% Solution in Carbon Disulfide
			Clay Remaining in Suspension	Hence Degree of Sedimentation	
		Per Cent	Per Cent	Per Cent	Per Cent
Trinidad residual asphalt...	50	33.5	33 7	0.0	2.23
Venezuela residual asphalt*.	48	32.4	30 1	7.0	1.91
Mexican residual asphalt...	50	33.3	27.2	18.3	1.89
California residual asphalt..	50	31.8	23 8	25.2	0.94
Mid-continental semi-asphaltic residual asphalt...	51	33 8	21.7	35.8	0.54

* Derived from Bababui heavy petroleum

Test 7g-2. Ultramicroscopic Count of Colloidal Particles. The ultramicroscope consists essentially of a compound microscope arranged for examining in a dark field an intense convergent beam of light cast upon the particles of the substance under examination, so that the latter will diffuse the light as viewed by the eye. An in-

tense, but minute beam of light is focused on the fluid contained on a glass slide or in a small cell, above which a compound microscope is adjusted vertically, so that the narrowest part of the light cone occupies the center of the focal field. If the fluid under examination is optically clear, or if it contains particles so minute that they cannot diffract sufficient light to create a visual impression, the light cone cannot be seen. If, however, enough light is diffracted by the particles, they become visible against a dark field. Particles may thus be observed that are beyond the resolving power of an ordinary microscope.

The following table will indicate the relative sizes of particles examined by various methods:

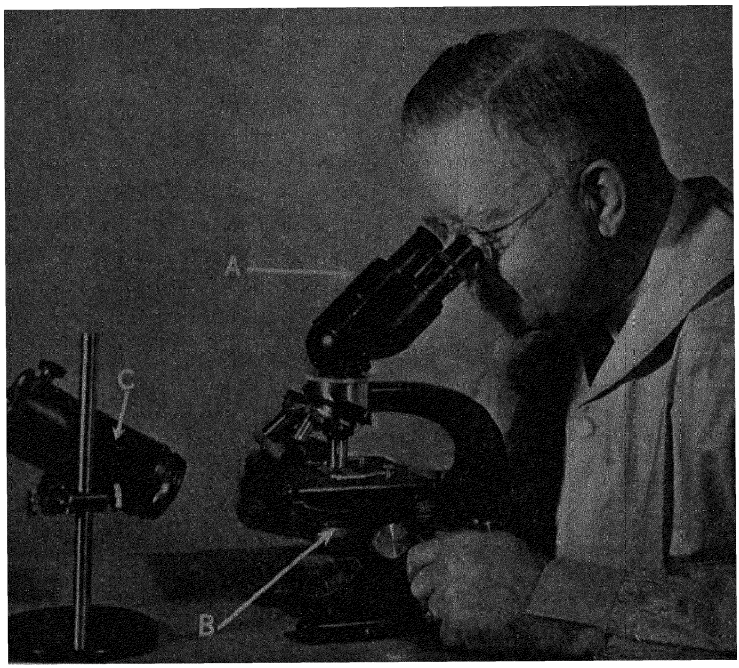
Material Unit	Diameter of Particles	Method of Examination
Atoms	0.1–2.0$\mu\mu$	X-ray spectrometer
Molecules	0.5–5.0$\mu\mu$	" "
Molecular groups	1 –10$\mu\mu$	Ultramicroscope
Primary colloidal particles	2 –20$\mu\mu$	"
Secondary colloidal particles	5 –100$\mu\mu$	"
Microscopically resolved particles	Over 250$\mu\mu$	Ordinary microscope
Particles visible to the eye	About 10μ	Unaided eye.

Note: 1μ =0.001 mm.–1 micron.
1$\mu\mu$ =0.001μ–0.000001 mm.

Particles retained by filter paper measure above 1μ; particles which pass through filter paper measure up to 100$\mu\mu$; particles beyond the resolving power of the ordinary microscope, but which are visible under the ultra-microscope (250$\mu\mu$) are termed "ultra-microns" or "sub-microns." If they cannot be discerned with the ultra-microscope, they are termed "amicrons." Rapid Brownian movement is observed when particles measure less than 10$\mu\mu$ in diameter; slow Brownian movement occurs with particles which measure between 10$\mu\mu$ and 1μ; and no Brownian movement occurs when the particles measure greater than 1μ.

The best solvents for use in dissolving bituminous substances in their ultramicroscopic examination are carbon disulfide and benzol, since these have about the same surface-tension as the substance itself, in the case of asphalts. Nitrobenzol is recommended for tars and pitches. The ultramicroscopic image of asphalts is not

easy to obtain, since the ultramicrons are very minute and not very luminous. A powerful source of light and painstaking focusing are required. Natural asphalts show bright particles of mineral matter, which are coarser than the ultramicrons of carbon present in petroleum asphalts. An 0.1 to 0.01 per cent solution of coal tar or coal-tar pitch in nitrobenzol gives clear images, showing many



Courtesy of Carl Zeiss. Inc.

FIG. 206.—Ultramicroscope with Dark Field Illuminator. A—High Powered Binocular Microscope; B—Sub-stage Dark-field Condenser; C—Source of Illumination.

particles in active Brownian movement. Such particles are not as luminous as those of natural asphalt, or mixtures of petroleum asphalt with fine inorganic fillers. Dark-field illuminators are fitted to the substage of the microscope in place of the usual Abbé substage condenser, and must be precisely centered with respect to the optical axis of the microscope. The usual arrangement is shown in Fig. 206.

The light beam should be introduced through an aperture of

1.00 to 1.40. The object should be covered with a thin cover-glass, which serves the purpose of totally reflecting the light beam. Oil or water immersion should be employed for the objective, which should have a minimum aperture of less than 0.8.[25]

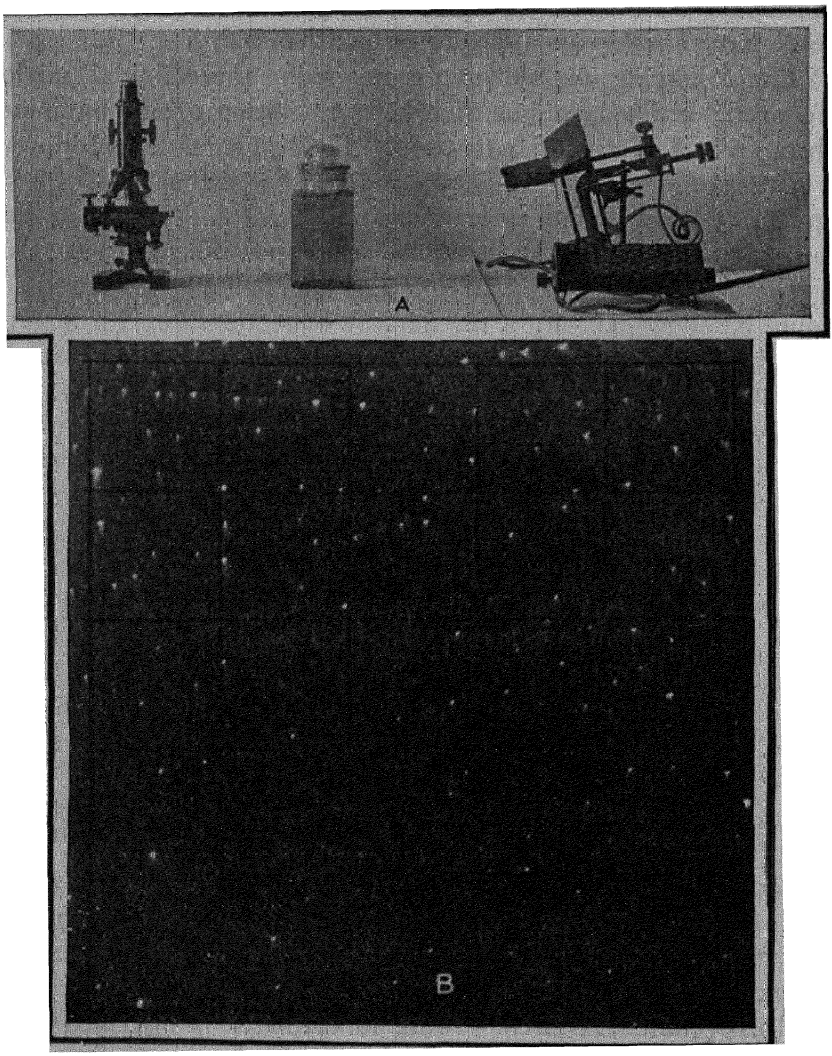
The following method involves counting the colloidal particles in a predetermined volume of a benzol solution of the bituminous substance at a dilution of 1 to 5000. The test is made as follows:[26]

Description of the Microscope. Use an ordinary microscope capable of giving a linear magnification of from 50 to 740 diameters at a tube-length of 160 mm. Use an eyepiece "× 7.5" and an objective "4 mm.," magnifying 320 diameters. The microscope should be equipped with a mechanical stage for lateral orientation as well as an accurately calibrated micrometer screw for recording vertical measurements. The eyepiece should carry a counting device consisting of a cross-line micrometer scale with a ground glass border divided into 25 squares, each side of which measures 1.25 mm. and corresponds to exactly 0.05 mm. of a stage micrometer at a tube-length of 160 mm. A ray of light from an arc lamp is passed through a cooling bath, acting as a ray filter, composed of 10 mgm. diamine green dissolved per liter of distilled water, and by means of a mirror at the base of the microscope projected through a substage paraboloid condenser having a central stop, which serves to bring the rays to a sharp focus. In this manner, the colloidal particles, whose indices of refraction vary from that of the enclosing liquid become luminously visible, whereas the remainder of the field remains perfectly dark. The microscope is set up as illustrated in Fig. 207.

Description of the Cell. The cell, illustrated in Fig. 208, consists of a 26 × 76 mm. object glass between 1.25 and 1.75 mm. thick, having a cavity excavated therein 2½ mm. in diameter and not exceeding 0.10 mm. deep with a corresponding capacity of less than 0.10 cmm. A circle 1 mm. in diameter is inscribed at the bottom of the cell in its center with a diamond-point marker. The cell and cover glass must be cleaned preparatory to filling, by boiling in concentrated sulfuric acid, rinsing in water, alcohol and benzol, drying with soft cotton cloth and rubbing with optical tissue paper.

Preparation of the Solutions. To prevent Brownian movement

of the particles, they are examined in a viscous solution of colloid-free asphaltic residual oil containing paraffin, prepared by fluxing



Courtesy E. C. E. Lord.

FIG. 207.—Microscope for Counting Colloidal Particles. A—Assembly of Apparatus. B—Appearance of Field Showing Colloidal Particles.

2.5 g. Mexican petroleum asphalt (penetration 148 at 77° F. with 0.5 g. crystalline paraffin, and then diluting to 100 ml. with benzol

containing 10 per cent ethyl alcohol. This is evaporated to constant weight on a water-bath, diluted to its original volume with benzol and passed several times through an alundum filter tube packed with macerated filter paper until not more than 10 particles are visible per ¼ mm. square, when examined in accordance with the method about to be described. Dissolve 1 g. of the specimen to be examined in 50 ml. benzol in a stoppered centrifugal tube; let stand overnight and then centrifuge for one hour at a speed of 800 revolutions per minute. With pipette draw off 1 cmm. of the liquid

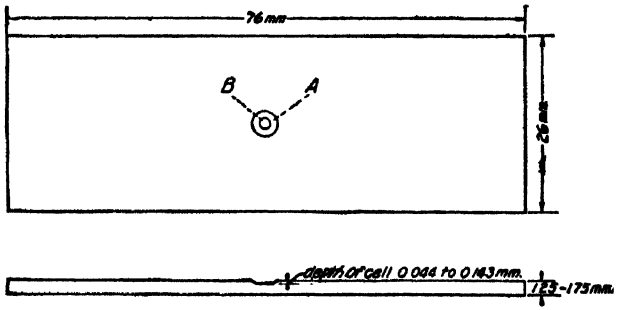


FIG. 208.—Cell for Counting Colloidal Particles.

at a depth of 10 mm., transfer to a glass-stoppered graduate, and dilute to 10 ml. with the paraffin-asphaltic oil dilutant. By this means a dilution of 1 to 5000 of the colloids present in the original sample is obtained.

Counting the Colloidal Particles. With a pipette, rapidly transfer 1 or 2 drops of the properly diluted solution to a cleaned cell and cover immediately with an 18-mm. cover glass, pressing it with a bluntly pointed wooden rod to expel the excess liquid and secure close contact. When the solution exuding from the cover glass hardens by evaporation, seal the cover glass with a 30 per cent solution of Canada balsam in ether. Transfer the slide to the microscope stage, and place upon it a few drops of an immersion liquid, such as cedar oil or glycerine to prevent loss of light through refraction of the rays issuing from the condenser. In correct focus, the colloidal particles will appear as brilliant points of light against a dark background as illustrated in Fig. 207(B). They should be

evenly distributed and in constant, though restricted motion. Examine the area within the central millimeter circle, representing exactly ¼ square mm., equivalent to four fields of the cross-line eyepiece-micrometer. Count the number of particles in each of the 25 square subdivisions of the micrometer, throughout the entire depth of liquid. From the average of four such determinations, compute the number of colloidal particles per cmm. of solution at 1 : 5000 dilution.

Results Recorded. Table LXXXVIII contains a summary of the observations made on some typical bituminous substances.

TABLE LXXXVIII

COLLOIDAL PARTICLES IN TYPICAL ASPHALTS

	Penetration at 77° F. (Test 9b)		Colloidal Particles	
	Before Treatment	After Treatment	Average per cmm. at 1 to 5000 Dilution	Relative Standing
(a) Refined Trinidad asphalt	2	.....	406,256	100 0
(b) Refined Trinidad asphalt combined with 33% colloidal clay (Note 1)	2	.....	30,529	7 5
Residual asphalt from Trinidad petroleum:				
(c) Combined with 33% clay (Note 2)	.....	235	169,262	41 6
(d) Heated to 340° F. with 16⅔% $CuCO_3 \cdot Cu(OH)_2$	.....	18	1,085,950	267 3
(e) Heated to 340° F. with 10% $CuCO_3 \cdot Cu(OH)_2$	.....	25	785,500	193 3
Residual asphalt from Mexican petroleum:				
(f) Combined with 33% clay (Note 2)	145	13	108,113	26 6
(g) Heated to 340° F. with 16⅔% $CuCO_3 \cdot Cu(OH)_2$	57	8	2,858,000	703 5
(h) Heated to 340° F. with 10% $CuCO_3 \cdot Cu(OH)_2$	57	10	1,012,800	249.3
(i) Heated to 340° F. with 20% $Pba_2 \cdot 3H_2O$	250	70	262,400	64.6
(j) Heated to 340° F. with 10% $CuSO_4$	57	16	204,160	50.2
(k) Heated to 340° F. with 10% $FeSO_4$	57	28	18,667	4.6
(l) Heated to 340° F. with 10% $ZnSO_4$	57	16	4,459	1 1
(m) Gilsonite heated to 340° F. with 10% $CuCO_3 \cdot Cu(OH)_2$	0	0	100,721	24 8
(n) Water-gas tar heated to 340° F. with 20% $CuCO_3 \cdot Cu(OH)_2$	.....	21	20,455	5.0

NOTE 1.—All insoluble organic and mineral matter removed before mixing with the clay (see Note 2), by dissolving in benzol, adding 2 per cent shellac dissolved in alcohol, evaporating to constant weight, redissolving in benzol, and finally filtering through an alundum tube, so the solution is essentially free from colloidal particles

NOTE 2.—Incorporated by Clifford Richardson's method of heating an aqueous emulsion of clay and asphalt, until all the moisture is expelled. See Test 7g-1.

The copper carbonate salts become largely reduced to red cuprous oxide, accompanied by an enormous colloidal dispersion, while with the remaining salts the reduction is much less complete (*i* and *j*) or entirely lacking (*k* and *l*) and the development of colloids correspondingly less. The accuracy of the method depends

chiefly upon the care exercised in the construction of the cell, as well as upon the proper consistency and optical purity of the diluting liquid.

The following method has been proposed for examining road tars:[27] Dissolve 0.2 to 0.3 g. of the tar in C.P. nitrobenzol (with the least possible agitation), so as to form exactly 1 volume per cent solution (having first ascertained the specific gravity of the substance). The solution is warmed on a water-bath to 25° C.; cooled to room temperature; then filtered through a 7-cm. Schleicher & Schüll No. 595 filter to remove the coarse particles. One drop of the well-shaken filtrate is transferred to an accurately scaled slide-glass (divided into squares 0.05 mm.—such as a Thoma hemocytometer) in a layer 0.1 mm. deep, and examined under an ultramicroscope under a magnification of × 400. After allowing the slide to stand 1–2 minutes, the "micella" are counted in various squares and the average computed. In making the count, any aggregation of particles are taken as a single particle. The average multiplied by 400,000 gives the number of micella per cubic millimeter of tar.

It is contended that the greater the number of micella, the better will be the binding power of the tar. In Holland (where this test originated), specifications call for a minimum of 10 million micella for good road tars. The number of micella is influenced by the age of the specimen, the temperature to which it has been heated, and the extent of mechanical manipulation (i.e., stirring or agitation). The foregoing test must therefore be performed under carefully controlled conditions.[28]

(B) MECHANICAL TESTS

VISCOSITY

This test is of value in determining the adaptability of the bituminous substance for a given purpose, for gauging the uniformity of supply, and for factory control work. It is used particularly for examining liquid to semi-liquid substances for road purposes, and may also be used to good advantage for predetermining the ability of semi-solid substances to saturate fabrics at elevated temperatures.

It has been observed that the viscosity of mixtures is invariably lower than an additive result of computations.[29] With most bituminous substances, the relation between viscosity and temperature may be expressed by a simple exponential function:

$$v \times t^n = a$$

where v = viscosity; t = temperature; and "n" also "a" represent two constants. If the logarithm of viscosity is plotted against the logarithm of temperature, a straight-line relationship will result. If the logarithm of the temperature is plotted as ordinate and the logarithm of the viscosity as abscissa, then the tangent of the angle which the straight line forms with the abscissa represents a measure of the change of viscosity with temperature. The susceptibility of viscosity to temperature changes may be computed from the expression:

$$\frac{\log v_1 - \log v_2}{\log t_2 - \log t_1}$$

where v represents the viscosity in poises and t represents the corresponding temperature in °C.[30]

It has also been proposed to determine the viscosity of tars and pitches in terms of the so-called "equi-viscous temperature," which represents the particular temperature at which the material will exhibit a predetermined viscosity.[31] For example, in using the B.R.T.A. viscosimeter as standardized by the Standardization of Tar Products Tests Committee of Great Britain [32] this temperature is one at which the tar attains a viscosity of exactly 50 seconds. The method consists in finding the viscosity of the material at two or more temperatures, then plotting the results logarithmically, and by interpolation, ascertaining the temperature at which the line crosses the "log. 50-sec." line. It has been reported that the range of commercial road tars is from 20° to 55° C., and for briquetting pitch in the neighborhood of 100° C. equi-viscous temperature. It has also been noted that the equi-viscous temperature of a mixture of the two tars corresponds closely to the mean of the equi-viscous temperature of the components; furthermore, that the reduction in

equi-viscous temperature of a tar caused by the addition of flux oil, is approximately proportional to the amount of oil added.

Test 8a. Engler Method. The Engler viscosimeter is illustrated in Fig. 209, and consists of a cylindrical vessel *a*, 105–6 mm. in diameter, with a cover *b* and a convex bottom, opening into a platinum-lined tube *c*, 20 mm. long, 2.9 mm. in diameter at the top, and 2.8 mm. at the bottom. The orifice may be opened or closed with the wooden plunger *d*. Metal projections are fastened to the inside of the vessel 25 mm. from the lowest portion of the cylindrical sidewalls and 32 mm. from the upper opening of the orifice. These serve to control the volume of bituminous liquid introduced, which amounts to exactly 240 ml. The bituminous material is maintained at any desired temperature by the heating-bath consisting of water, glycerine or cotton-seed oil in the cylindrical jacket *f*, heated by the ring-burner *h*, and the temperature recorded by the thermometer *e*.[33] The output of 200 ml. of distilled water at 68° F. is carefully observed. If the apparatus has been constructed properly, this will require 50–52 seconds.

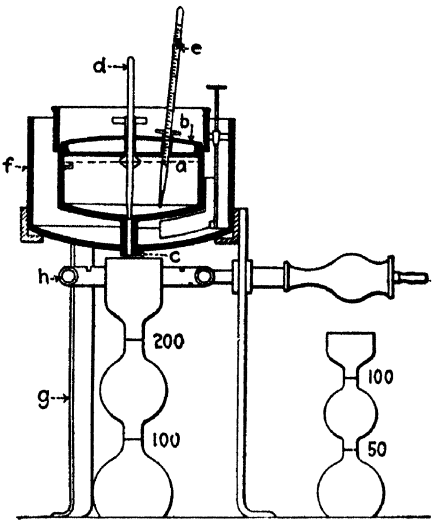


FIG. 209.—Engler Viscosimeter.

The bituminous material is ordinarily tested at 77° F. (25° C.), 172° F. (50° C.), or 212° F. (100° C.) depending upon its consistency. The viscosimeter is filled to the top of the points with bituminous material brought to the required temperature, and the time noted for 200, 100, 50 or 20 ml. to flow through the orifice. If 100 ml. are allowed to flow through the instrument, the reading should be multiplied by 2.35 to calculate the time of flow for 200 ml. If 50 ml. are allowed to flow through, the reading should be multiplied by 5, and with 20 ml. by 11.95 to obtain the time of flow for 200 ml.

If insufficient material is available to fill the viscosimeter, either 25 or 45 ml. may be introduced, and 10 or 20 ml. respectively al-

lowed to flow through, in which events the readings should be multiplied by 13 or 7.25 respectively to obtain the time of flow for 200 ml. These factors are constant. The specific viscosity at $t°$ F. compared with water at 68° F. is equal to the number of seconds for 200 ml. of the substance to pass through at $t°$ F. divided by the seconds for 200 ml. of water to pass through at 68° F. Tables have been worked out showing the factor to be used when the apparatus is filled with smaller volumes of liquid allowing different amounts to flow through.[34] For testing roofing saturants, the time of flow of 50 ml. at 350° F. should be recorded.

Test 8b. Saybolt (Furol) Method. This test has been standardized as follows:[35]

The Saybolt Universal Viscosimeter shall be used only for substances with times of flow of more than 32 sec. There is no maximum limit to viscosity to be measured by the Saybolt Universal Viscosimeter but, in general, liquids having an outflow of the order of 1000 sec. and higher, Saybolt Universal, are tested more conveniently by means of the Saybolt Furol Viscosimeter.

The Saybolt Furol Viscosimeter shall be used only for substances with times of flow of more than 25 sec. The outflow time of the Furol (Note) instrument is approximately one-tenth that of the Universal.

NOTE.—The word "Furol" is a contraction of the phrase "fuel and road oils."

The apparatus shall consist of an oil tube, bath, receiver, thermometers, timer, and withdrawal tube, conforming to the requirements specified in the following Paragraphs (*a*) to (*g*):

(*a*) *Oil Tube.*—The oil tube (illustrated in Fig. 210A) shall be entirely of corrosion-resistant metal, with or without plating, and shall conform to the dimensional requirements shown in Table LXXXIX within the permissible variations prescribed, The lower end of the oil tube shall be provided with a nut for locking it in place in the bath and with a cork or other suitable device to prevent flow until the test is started. For convenience a string or its equivalent may be attached to the cork for rapid removal.

The oil tube shall be standardized, and any correction in excess of 0.2 per cent shall be applied. The time of flow shall be within ± 1 per cent of the time as obtained with the National Bureau of Standards' master Saybolt oil tube.

TABLE LXXXIX

DIMENSIONS OF OIL TUBES

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furol Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm	Normal, cm	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0 1780	0 313	0 315	0.317
Outside diameter of outlet tube at lower end	0 28	0 30	0 32	0 40	0 43	0.46
Length of outlet tube*	1 215	1 225	1 235	1 215	1 225	1.235
Height of overflow rim above bottom of outlet tube*	12 40	12 50	12.60	12.40	12 50	12.60
Outside diameter of overflow rim, at the top*,‡	†		3 30	†		3 30
Diameter of container*	2.955	2 975	2.995	2 955	2 975	2.995
Depth of cylindrical part of container*	8.8			8.8		
Diameter of container between bottom of cylindrical part of container and top of outlet tube*	0 9			0 9		

* This dimension is identical in the Saybolt Universal and the Saybolt Furol instruments.

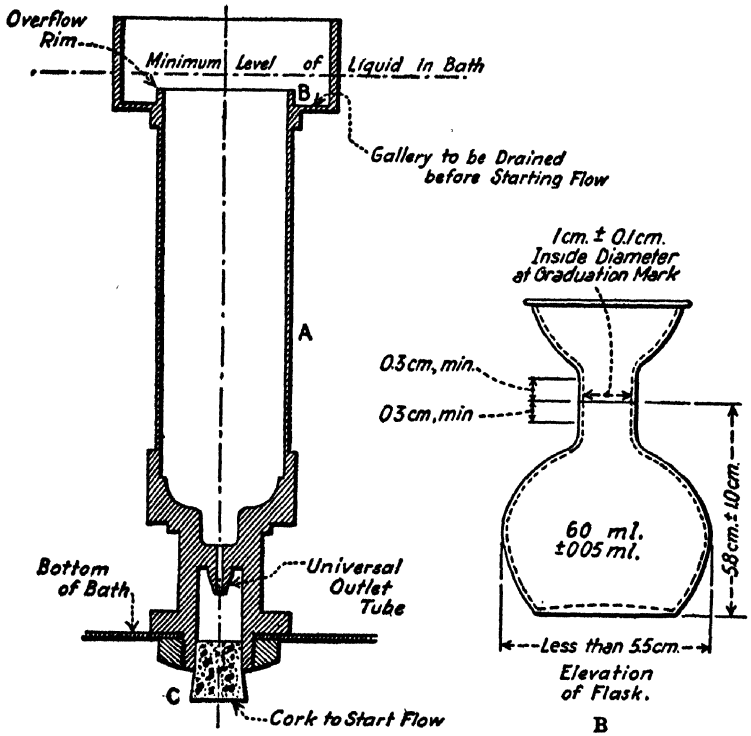
† The minimum value shall preferably be not less than 3.2 cm.

‡ The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

(*b*) *Bath.*—The bath shall serve as a support to hold the oil-tube in a vertical position and as a container for the bath liquid. The bath shall be equipped with a stirring device and with means for heating or cooling. The source of heat or refrigeration shall be more than 1¼ in. (3 cm.) from the oil tube. If an external heater is used, it must be more than 2 in. (5 cm.) from the oil tube. The bath temperature necessary to maintain thermal equilibrium (while the oil in the tube is well stirred by the oil-tube thermometer) shall be within ± 0.1° F. (0.06° C.) of the standard temperatures of 70° F. (21.1° C.) or 77° F. (25° C.), or shall not exceed 100.25°, 122.35°, 130.5°, 141.0°, 181.5° or 212.0° F. (37.9°, 50.2°, 54.7°, 60.6°, 83.1° or 100.0° C.), respectively, for the standard temperatures mentioned below. The level of the bath liquid shall not be lower than ¼ in. (0.5 cm.) above the overflow rim of the oil tube. For referee testing, the bath liquid must be one which in the bath used will meet the preceding bath-temperature conditions (see Note).

NOTE.—These temperature requirements can be met with water, aqueous solutions, and some baths with oil. In routine testing oil is generally used as the bath medium. This is allowable provided the temperature of the oil bath is adjusted so that the necessary condition of thermal equilibrium is maintained. It may be necessary to maintain the oil bath at slightly higher temperatures than those specified in the preceding paragraph. Temperature differentials between oil bath and oil tube necessary to maintain thermal equilibrium may be double those given above.

(*c*) *Receiver.*—The receiving flask (see Fig. 210B) shall be of glass with a capacity up to the graduation mark on its neck of 60 ml. ± 0.05 ml. at 68° F. (20° C.). At the graduation mark, the inside diameter of the neck of the flask shall be 1 cm. ± 0.1 cm. The cylindrical portion of the neck of the flask shall extend not less than 0.3 cm. above and below the graduation mark. The graduation mark shall be 5.8 cm. ± 1.0 cm. above the bottom of the flask. The maximum outside diameter shall be less than 5.5 cm.



Courtesy A.S.T.M.

FIG. 210.—Saybolt Viscosimeter.

(*d*) *Oil-Tube Thermometers.*—The oil-tube thermometers shall conform to the following requirements. They shall include two sets of six thermometers each, one set being graduated in Fahrenheit degrees and the other in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing. To prevent contact of the thermometer with the orifice in the oil tube a suitable support shall be attached to the enlargement of the thermometer stem.

(*e*) *Bath Thermometers.*—Oil-tube thermometers, or other means for measuring temperature of at least equal accuracy, shall be used in the bath.

(*f*) *Timer.*—The stop watch or other timing device used shall be graduated in divisions of 0.2 sec. or less, and shall be accurate to within 0.1 per cent when tested over a 60-min. period (see Note).

NOTE.—Electrical timing devices are permissible provided they are accurate and capable of being read to 0.2 sec. Timing devices actuated by synchronous motors shall be used only on electric circuits of controlled frequency.

(*g*) *Withdrawal Tube.*—The tube or pipette used for draining the gallery shall have a smooth tip of about 3 mm. outside diameter and about 2 mm. inside diameter.

With the Saybolt Universal Viscosimeter, determinations shall be made at 70°, 100°, 130° or 210° F. (21.1°, 37.8°, 54.4° or 98.9° C.).

With the Saybolt Furol Viscosimeter, determinations shall be made at 77°, 100°, 122° or 210° F. (25°, 37.8°, 50° or 98.9° C.). In tests on road and paving materials, determinations may also be made at 140° and 180° F. (60° and 82.2° C.).

Viscosity determinations shall be made in a room free from drafts and rapid changes in temperature. For standardization and referee tests the room shall be between 68° and 86° F. (20° and 30° C.) and the actual temperature shall be recorded. For routine tests, temperatures up to 100° F. may be employed without introducing errors in excess of 1 per cent. Determinations shall not be made at temperatures below the dew point of the atmosphere surrounding the instrument. The oil tube shall first be cleaned with an effective solvent, such as benzol, and excess solvent shall be removed from the gallery.

All oil shall be passed through a 100-mesh wire strainer before it is introduced into the oil tube. After the tube is cleaned, a quantity of the oil to be tested, sufficient to wet the entire surface of the tube, shall be poured into the tube and allowed to drain out (see Note). The cork stopper shall be inserted not less than ¼ in. nor more than ⅜ in. into the lower end of the air chamber at the bottom of the oil tube. The cork shall fit tightly enough to prevent the escape of air, as evidenced by the absence of oil on the cork after it is withdrawn.

NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards.

If the test temperature is above that of the room, the oil shall

be heated to not more than 3° F. (1.7° C.) above the temperature of test and if the temperature is below that of the room, the oil shall be cooled to not more than 3° F. (1.7° C.) below the temperature of test. The oil shall be poured into the oil tube until it ceases to overflow into the gallery. The oil in the oil tube shall be kept well stirred with the oil-tube thermometer, care being taken to avoid hitting the outflow tube. The bath temperature shall be adjusted until the oil temperature remains constant. After thermal equilibrium has been attained, no further adjustments shall be made in the bath temperature. The test result shall be discarded if the indicated bath temperature varies by more than ± 0.05° F. (0.03° C.) in tests at 70°, 77°, 100°, 122° and 130° F. (21.1°, 25°, 37.8°, 50° and 54.4° C.), or by more than ± 0.10° F. (0.06° C.) in tests at 140°, 180° and 210° F. (60°, 82.2° and 98.9° C.).

After the temperature of the oil in the oil tube has remained constant within 0.02° F. (0.01° C.) of the desired temperature for 1 min. with constant stirring, the oil-tube thermometer shall be withdrawn and the surplus oil removed quickly from the gallery by means of the withdrawal tube, so that the level of the oil in the gallery is below the level of the oil tube proper. The tip of the withdrawal tube shall be inserted at one point in the gallery. The test shall be started over again if the tip of the withdrawal tube touches the overflow rim. Under no conditions shall the excess oil be removed by rotating the withdrawal tube around the gallery.

The receiving flask shall be placed in position, so that the stream of oil from the outlet tube will strike the neck of the flask. The graduation mark on the receiving flask shall be not less than 10 cm. or more than 13 cm. from the bottom of the bath. The cork shall be snapped from its position and at the same instant the timer shall be started. The timer shall be stopped when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

The time in seconds as determined by the prescribed procedure, with the proper calibration correction, is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test is made. Results shall be reported to the nearest 0.1 sec. for viscosity values below 200 sec. and to the nearest whole second for values 200 sec. or above. With proper attention to details of pro-

cedure, results in different laboratories with different operators under referee or standardization conditions of testing, should not differ by more than 0.5 per cent.

It has been reported that the Saybolt-Furol viscosity at any particular temperature is approximately 4 times the Engler specific viscosity at the same temperature.

Test 8c. Absolute Viscosity. The "poise" is the unit used to measure the absolute viscosity of fluids. When a fluid fills the space between two planes 1 cm. apart, each measuring 1 sq. cm., the tangential force in dynes which must be exerted to move one of the planes parallel to the other, exactly 1 cm. in 1 sec., represents the absolute viscosity of the fluid in poises (μ). A simple instrument has been described for ascertaining the absolute viscosity of bituminous substances by means of a graduated capillary tube to which suction is applied.[36]

Fig. 211A shows the viscosimeter proper in detail, and Fig. 211B the schematic arrangement of the apparatus when assembled. Approximately 5 cm. of the substance are placed in the sample-cup, the upper level of which should fall ± 2 mm. from the 10 mm. graduation on the capillary tube, representing a depth of immersion approximately 1.0 cm.. When the viscosimeter and contents has been brought to the desired temperature, the vacuum reservoir is evacuated to an extent (measured in cms. of mercury) sufficient to cause the material under test to rise in the capillary at a speed neither too rapid nor unnecessarily slow. Open the stop-cock in the line, and with a stop-watch determine the number of seconds required for the interface to pass between two predetermined marks on the graduated capillary.

For fluid substances, start at the 40 mm. and end at the 120 mm. mark, using the following formula:

$$\mu = (25.909h - 17.163)\ r^2t.$$

For more viscous substances, start at the 20 mm. and end at the 40 mm. mark, using the following formula:

$$\mu = (20.4h)\ r^2t.$$

Where μ = absolute viscosity in poises; h = vacuum in cms. of mer-

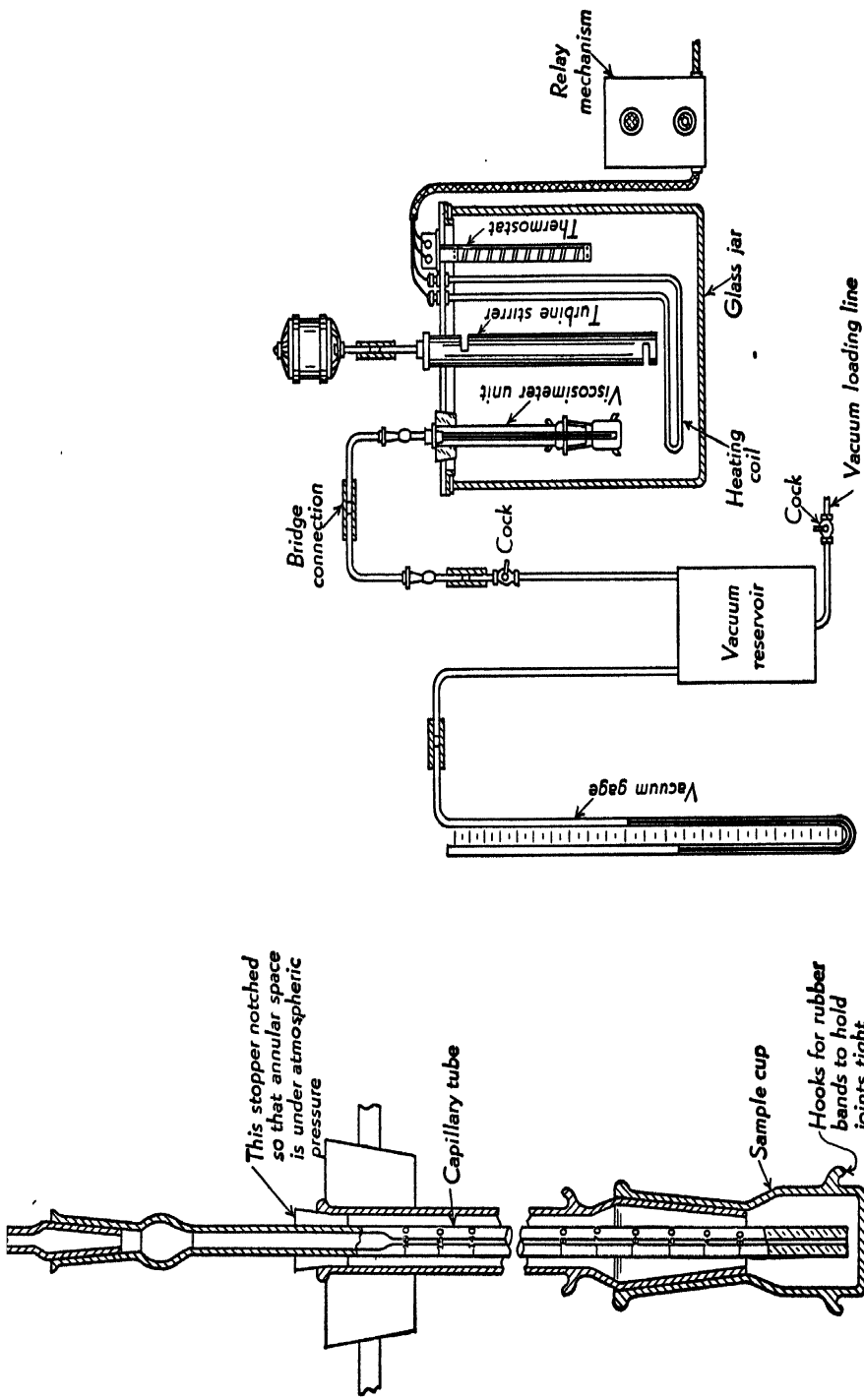


FIG. 211.—Apparatus for Determining Absolute Viscosity to Poises.
A—Cross section of Capillary-tube and Sample-cup; B—Assembly of Apparatus.

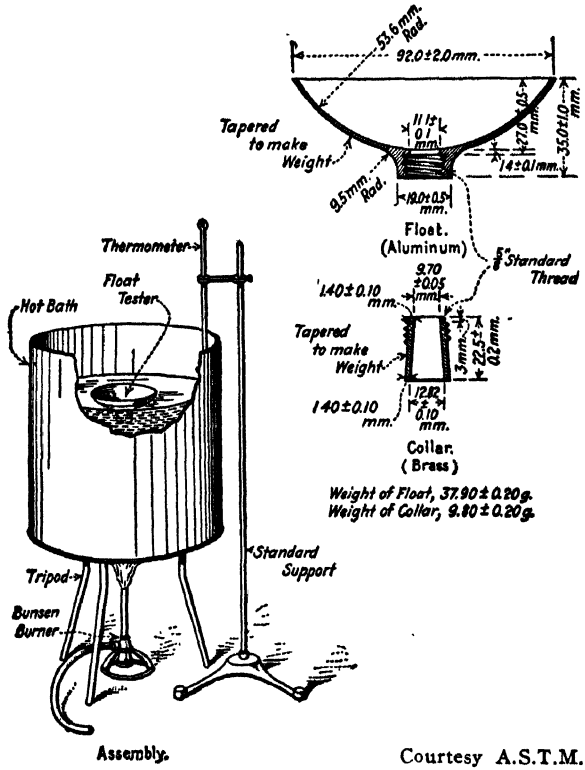
cury; r = radius of the capillary bore in cms.; and t = time of rise in seconds.

At the close of the test, the capillary tube may be cleansed with hot nitrobenzene, followed by acetone.

Test 8cc. Hutchinson's Method. This is illustrated in Fig. 212. It was invented by John Hutchinson [87] and consists of a metal



FIG. 212.—Hutchinson's Tar Tester.



Courtesy A.S.T.M.

FIG. 213.—Float Test Apparatus.

spindle 9 in. long over all, bearing a conical-shaped disc (C) 2 in. in diameter, midway between the ends, with a plumb-shaped weight fastened to its lower end. The instrument is supplied with three weights (D) to be used with tars of different consistency or gravity. The spindle bears two rings (A) and (B), 2 in. apart.

The test is conducted by placing the bituminous material in a cylinder at least 9 in. high and 4 in. in diameter, filled to ½ in. of the top. The bituminous material is brought to exactly 77° F., the tester

introduced, and the time in seconds is noted for the spindle to sink from *a* to *b*. Weight No. 1 is recommended for tars having a specific gravity of 1.170–1.195, No. 2 from 1.195–1.215, and No. 3 from 1.215–1.240. The weights do not conform to standard weights or dimensions, and the instrument should not therefore be regarded as a strictly scientific one. It is used extensively in England.

Formulas and monograms have been worked out, showing the relation between the consistency and temperature of tars using the Hutchinson tester,[38] also the relation between the consistency of tars and petroleum asphalts in various admixtures.[39]

Test 8d. Float Test. This instrument is used largely for testing the viscosity or consistency of semi-solid bituminous materials. The range of the float test is limited, and it cannot be used with very fluid bituminous materials or with hard solids. It accordingly fills the gap between the Engler viscosimeter on one hand, and the needle penetrometer and consistometer on the other. The test is not affected by the presence of finely divided mineral matter or free carbon.

The procedure has been standardized as follows:[40]

(*a*) The float (Fig. 213) shall be made of aluminium or aluminium alloy and shall be in accordance with the following requirements:

	Minimum	Normal	Maximum
Weight of float, g	37.70	37.90	38.10
Total height of float, mm	34.0	35.0	36.0
Height of rim above lower side of shoulder, mm	26.5	27.0	27.5
Thickness of shoulder, mm	1.3	1.4	1.5
Diameter of opening, mm	11.0	11.1	11.2

(*b*) The collar shall be made of brass and shall be in accordance with the following requirements:

	Minimum	Normal	Maximum
Weight of collar, g	9.60	9.80	10.00
Over-all height of collar, mm	22.3	22.5	22.7
Inside diameter at bottom, mm	12.72	12.82	12.92
Inside diameter at top, mm	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 213. The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being —2 to +80° C. or +30 to +180° F., respectively. The diameter of the bath and the depth of water shall be at least 185 mm.

The brass collar shall be placed with the smaller end on a brass plate which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury.

The sample shall be completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote oil residues, which shall be mixed and poured at a temperature of 100 to 125° C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

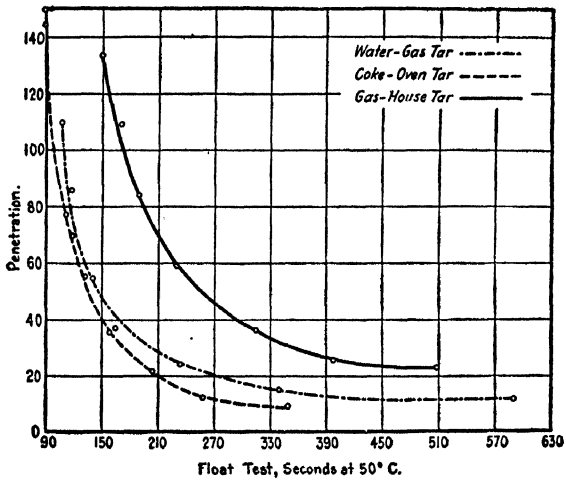
Asphalt and Asphalt Products. Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5° C. for five minutes, after which the surplus material shall be removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ± 1° C., and left in this bath for at least fifteen minutes.

Tar Products. Tar products shall be immediately immersed in ice water maintained at 5° C. for five minutes, after which the surplus material shall be removed by means of a spatula or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., ± 1° C., and left in this bath for at least fifteen minutes.

The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature shall be accurately maintained and shall at no time throughout the

test be allowed to vary more than 0.5° C. from the temperature specified.

After the material to be tested has been kept in the ice water for not less than fifteen minutes nor more than thirty minutes, the collar with its contents shall be removed from the plate and screwed into the aluminium float and immersed in water at 5° C. for one minute. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and



Courtesy A.S.T.M.

FIG. 214.—Comparison of the Float and Penetration Tests for Different Types of Tars.

out of the collar until the water gains entrance into the saucer and causes it to sink.

The time in seconds between placing the apparatus on the water and when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precautions should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

Figure 214 shows the relation between the float test and penetration test (200 g., 5 sec., 32° F.) of three types of tars, all of which were evaporated and samples taken having softening points

of 95, 100, 105, 110, 115, 120 and 125° F. respectively, cube-in-water method:[41]

The author has also found the float test of value for testing bituminous substances at a temperature *exactly* 50° F. above the fusing-point by the R. and B. method, thereby furnishing a criterion of the susceptibility to temperature changes, also a means of distinguishing between blown and residual asphalts.

Test 8e. Schutte Method. This instrument, as illustrated in Fig. 215, operates similarly to the float tester, with the difference, however, that the pressure is applied by a column of water above the plug of pitch. The melted bituminous material is first introduced into a brass collar 1 in. high and 5/8 in. in diameter. This is placed in water at the required temperature for at least ten minutes, and then screwed into the tube (10 1/8 in. long). The apparatus is immersed in water maintained at the required temperature so the water level just covers the lower shoulder of the tube, which is then completely filled with water at the given temperature, and the time interval recorded between the filling of the tube and the displacement of the plug of bituminous material at the bottom. Check tests are said to agree within five seconds.[42]



Courtesy of A. H. Thomas Co.

FIG. 215.—Schutte Viscosity Tester.

Test 8f. Falling Ball Method. An electrical method has been devised [43] for recording the time in which a spherical steel ball falls a definite distance through the material at a given temperature, which is accomplished by noting the alterations in inductance and capacity of a closed oscillatory circuit of 31.5 megacycles, by the passage of the ball through two "search coils" in the circuit, suspended at suitable positions in the material under examination. This method has been shown to yield scientifically correct results in absolute units, and will indicate whether or not the substance is a true liquid at a given temperature.[44] The apparatus is illustrated diagrammatically in Fig. 216, in which "b" represent an oil-bath; "V" the viscosimeter tube composed of an aluminium alloy, of an in-

ternal diameter sufficient to avoid "wall effect" when using the largest ball (½-in. diam.); "C" the search coils spaced exactly 10 cm. apart (center to center), the upper coil being approximately 10 cm. from the surface of the liquid and the lower coil approximately 10 cm. from the bottom of the viscosimeter tube; "H" the entrance hole for introducing the spherical ball; "T" the terminals; and "S" the stirrer. The search coils are wound in units of about 1-in. diameter and 7/32-in. long, and are mounted on a skeleton copper tube frame through which the lead wires are passed. They are wound in thin paper, thoroughly coated with shellac and baked. Audible indication of the passage of the falling ball through the search coils is obtained by coupling the circuit to a similar circuit tuned to produce audible notes on a telephone receiver. Balls ranging in diameter from 1/16 in. to ½ in. may be used and tests may be carried out at any desired temperature.

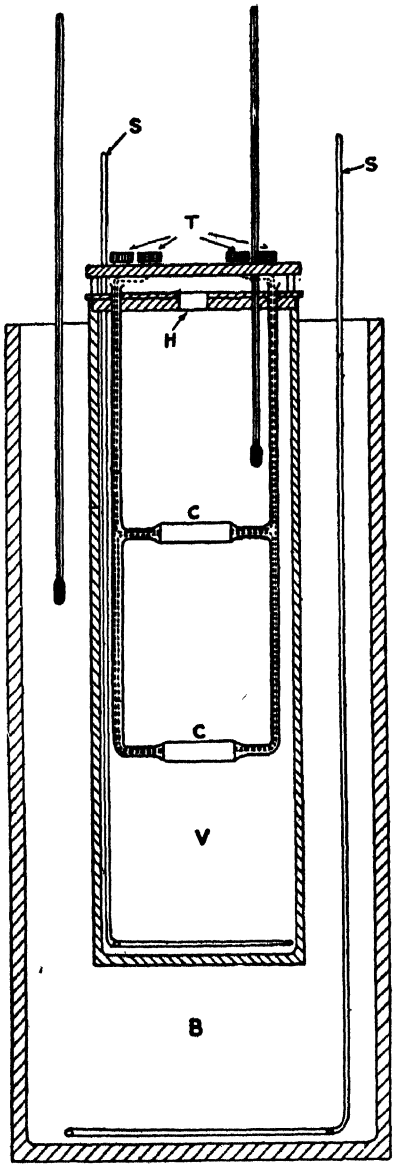


FIG. 216.—Falling-ball Viscosimeter.

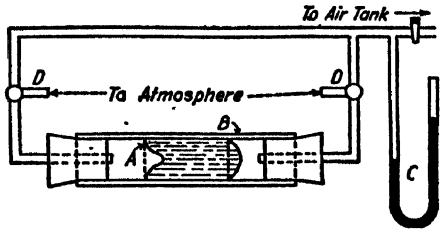
The viscosity in poises may be calculated from the following formula:

$$\eta = \frac{td^2(S - s)g}{18c\ (1 - 2.4d/D)\ (1 - 5d/3h)}$$

where: η = absolute viscosity in poises; D = diameter of the viscosimeter tube; d = diameter of the sphere; S = density of the sphere in grams per ml.; s = density of the substance in grams per ml.; c = distance in cm. between the two search coils; h = height of the test column of substance in cm.; t = time in seconds for the

sphere to drop the distance "c" between the two coils; $g = 981$ cm./sec.^{2}

Test 8g. Alternating Stress Method. This procedure is adapted to testing the viscosity of semi-solid bituminous substances in absolute units [45] ranging from about 1,000,000 to 100,000,000 poises. The apparatus is illustrated diagrammatically in Fig. 217, and consists of a cylinder "A" of the material, about 5 cm. long, contained in a glass tube "B" which may range in diameter from about 0.25 to 2.00 cm., connected to a tank of compressed air and a manometer "C" by three-way stopcocks "D," so that pressure may be applied to either end of the cylinder of bituminous material. The material is introduced in the tube "B" while held in a vertical position with an amalgamated brass plunger having a flat end inserted part way into the tube. The melted substance is then poured into the tube and onto the plunger, and upon cooling, both ends of the plug are flattened by manipulation with the amalgamated plunger. The tube and its contents are placed in a constant temperature bath so that the ends of the cylinder "A" may be observed by means of a cathetometer. The cathetometer is focused on one end of the cylinder and measured air pressure applied to the other end. An extrusion of the material occurs, the rate of movement being measured by the cathetometer. When the extrusion takes place to a distance equal to one-half of the radius of the tube, it is forced back by reversing the direction of the air pressure. At first, high pressures are applied and each succeeding pair of determinations is made at progressively lower pressure until a constant reading is obtained. If the substance is elastic or thixotropic, the sample must be worked back and forth several times before a reading is taken. Viscosity in poises may be calculated from the formula:



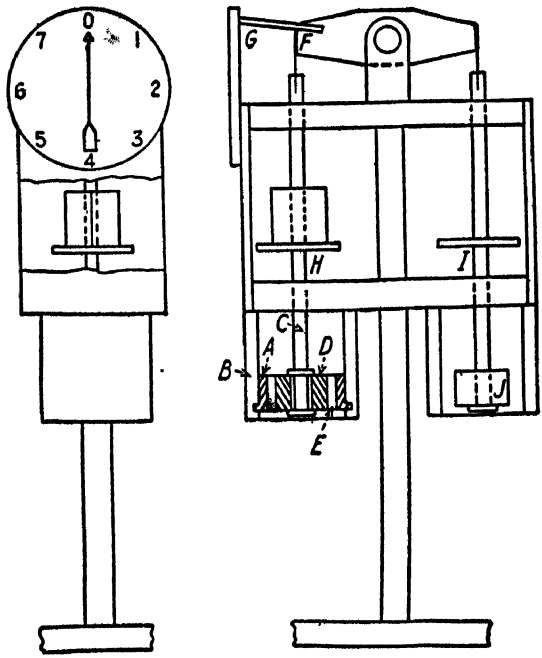
Courtesy A.S.T.M.

FIG. 217.—Apparatus Used in Alternating Stress Method.

$$\eta = \frac{PR^2t}{4Lh}$$

where: η = viscosity in poises; P = pressure applied in dynes per sq. cm.; R = radius of the tube in cm.; t = time of flow in seconds; L = length of the cylinder in cm.; and h = length of extrusion in cm., in the time "t."

Test 8h. Falling Coaxial Cylinder Method. This method is likewise adapted to measuring the viscosity of semi-solid bituminous substances ranging from 5,000 to 100,000,000 poises [46] and is a modification of the rotating cylinder method.[47] A schematic diagram of the apparatus is illustrated in Fig. 218, and consists of a



Courtesy A.S.T.M.

FIG. 218.—Falling Coaxial Cylinder Method.

brass outer cylinder "A" measuring ¾ in. in diameter; held in a bakelite holder "B" by arrangement which permits rapid insertion or removal of the mold. A bakelite rod "C" passes through the inner cylinder "D" measuring ½ in. in diameter and 1 in. in length. The material under test fills the annulus "E" measuring 16 ml. in volume. The bakelite rod "C" is connected to a metal ribbon which passes over a rocker-arm "F," which in turn is attached to another rod "I" and a counterpoise "J." A weight "H" applied to the rod

"C" causes the inner cylinder "D" of the viscosimeter to move downward; while added to rod "I" it causes cylinder "D" to move upward, the distance of movement of the inner cylinder in a given time being indicated on the dial "G." The apparatus is equipped with a series of weights to measure a wide range of viscosities. A thermostatically controlled bath surrounds the coaxial cylinders and the holder "B." Rod "C" and holder "B" are made of a non-metallic material to eliminate the conduction of heat away from the sample. The two cylinders "B" and "D" are heated and placed on an amalgamated brass plate which is provided with pins to center both cylinders, whereupon the melted substance is poured into the annular space, and when cool the base plate is removed and the apparatus assembled. After the inner cylinder has moved a predetermined distance, as measured by a cathetometer, the apparatus is reversed and the inner cylinder allowed to return to its original position. A test usually consists of three "out" and three "in" readings. The viscosity is calculated from the following formula:

$$\eta = \frac{gt}{2\pi Lh}\left[\log_e \frac{R}{r}(W - \pi r^2 LD) + \frac{\pi LD}{2}(R^2 - r^2)\right]$$

where η = viscosity in poises,
g = gravitation constant,
L = length of outer cylinder in centimeters,
h = distance in centimeters of movement in time t (sec.),
R = inner radius of outer cylinder in centimeters,
r = radius of inner cylinder in centimeters,
W = effective weight in grams of applied load (actual weight minus buoyant effect of liquid bath), and
D = difference in density of material being measured and density of bath in which the apparatus is run in grams per cubic centimeters.

If the bath in which the viscosimeter is immersed contains a liquid of substantially the same density as the substance being tested, the equation simplifies to:

$$\eta = \left(\log_e \frac{R}{r}\right)\left(\frac{g}{2\pi L}\right)\left(\frac{Wt}{h}\right) = KW\frac{t}{h}$$

where K is constant for a particular instrument. For thixotropic materials, the first "out" reading will give an abnormally low read-

ing, but all subsequent readings will approach a constant value. On the other hand, if the material is highly elastic, the initial rate of movement when the direction of flow is reversed, will be high. These low and high readings should be discarded when calculating the viscosity of the material.

HARDNESS OR PLASTICITY

This constitutes one of the most important tests for examining bituminous materials, and is employed for purposes of identification, considered either alone or in conjunction with the fusing-point; for determining the adaptability of bituminous materials in connection with certain proposed uses; for gauging the uniformity of supply; and for purposes of factory control.

The terms "consistency" and "plasticity" have been defined as follows:[48]

Consistency.—That property of a material other than a gas by virtue of which it resists the stresses which produce permanent change of shape.

NOTE 1.—Consistency is expressed by the three following flow-force relations:

Simple (Newtonian) Liquid.—When the ratio of flow to force is constant the material is said to be a simple liquid.

The simple liquid under conditions of non-turbulent flow follows Newton's law, which may be written, $v = \phi F r$, where v is the velocity imparted by the stress F per unit area, to either of two parallel planes separated by the distance r, the space between the planes being filled with the liquid. The constant ϕ is known as the *fluidity* which is the reciprocal of the *viscosity*.

Solid.—Any body which requires a finite stress to produce a permanent deformation is a solid.

Non-Newtonian Liquid.—A body which yields permanently under very small stresses and yet is not a simple liquid is known as a non-Newtonian liquid.

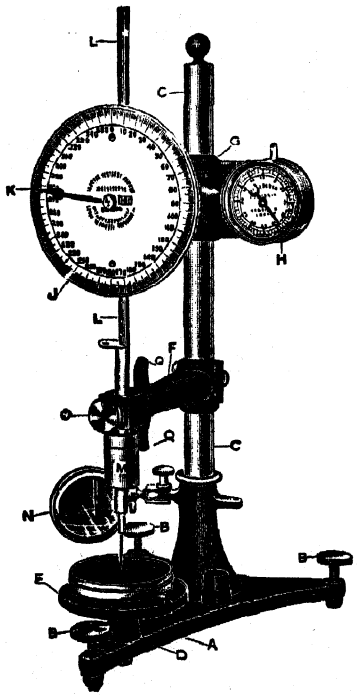
NOTE 2.—The term consistency may be used irrespective of whether the body is a liquid or a solid.

Plasticity.—That property of a solid by virtue of which it is permanently deformed when stressed above the yield value. This is a complex property and therefore cannot be represented quantitatively by a single numerical value. The factors which define plasticity are the yield value and the mobility:

Yield Value, θ.—The minimum stress required to produce a permanent deformation in a solid.

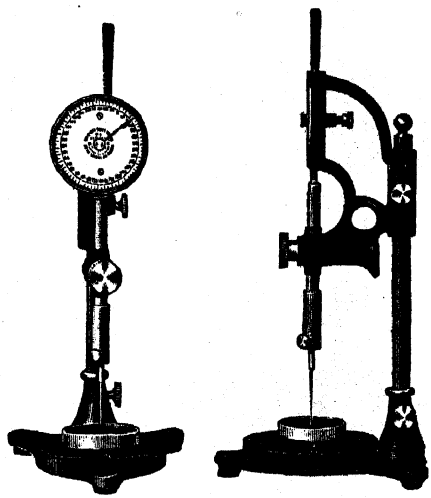
Mobility, μ.—The coefficient in the law of plastic flow, $v = \mu (F - \theta) r$, where v is the velocity imparted by the stress F per unit area, to either of two parallel planes separated by the distance r, the space between the planes being filled with the plastic material.

Test 9a. Moh's Scale. This test has long been used for recording the hardness of minerals by comparing their resistance to abrasion with substances of known hardness. Ten minerals are used in a graduated scale of units, viz.: (1) talc, (2) gypsum, (3) calcite, (4) fluorite, (5) apatite, (6) orthoclase, (7) quartz, (8) topaz, (9) sapphire and (10) diamond. A pointed fragment of the standard mineral is moved back and forth several times on the



Courtesy of Howard & Morse.

FIG. 219.—Standard Penetrometer.



Courtesy of Howard & Morse.

FIG. 220.—Miniature Penetrometer.

same line, a short distance across the surface of the bituminous material under test. If the bituminous material is not scratched, it is harder than the mineral used, whereas if it is scratched, it may be either softer or of the same hardness as the standard mineral. If it is of the same hardness, it will in turn scratch the surface of the standard mineral but if it is softer, it will have no effect. The first four standard minerals are used for this purpose, as the hardest bituminous materials encountered usually do not test higher than 4 on Moh's scale.

Test 9b. Penetrometer. This was originally devised by H. C. Bowen in 1888.[49] The first crude instrument was further improved by A. W. Dow.[50] The Dow penetrometer as simplified in construction by Clifford Richardson and C. N. Forrest represents the type in use today,[51] both forms operating on the same principle and giving the same readings.

The Richardson-Forrest improved penetrometer is illustrated in Fig. 219. The base *A* may be levelled by the thumb-screws *B*, and is attached to the standard *C* and also the platen *D*, which by means of a screw-shank raises or lowers the revolving disc *E*, on which is placed the sample of bituminous material to be tested. The standard *C* carries a bracket *F* adjustable as to elevation by a thumb-screw, also the bracket *G*, which on the back carries the clockwork *H* timing the duration of the test by half-second beats, and on the front the dial *J* divided into 360 degrees, with the hand *K*, marking the number of degrees, each of which represents one-tenth millimeter of penetration measured by rack on sliding gauge *L*, engaging in pinion on the shaft which actuates the hand *K*. The bevelled-edge mirror *N* adjustable through universal joints, serves to reflect light on the sample under test. The plunger *O* acts as a brake, which holds the needle bar, representing a weight of 50 g. together with the superincumbent weight in place, until pressed inward, which movement permits the needle and weight to act upon test-block without friction, and is easily operated by grasping the horns *Q* between two fingers and pressing the brake-head *O* with the thumb. *M* represents a weight of predetermined capacity, either 50 or 150 g. Forms of penetrometer operated by automatic timing devices have also been constructed.[52] A miniature penetrometer for portable use is illustrated in Fig. 220, and a form with a micrometer measuring device is illustrated in Fig. 221.

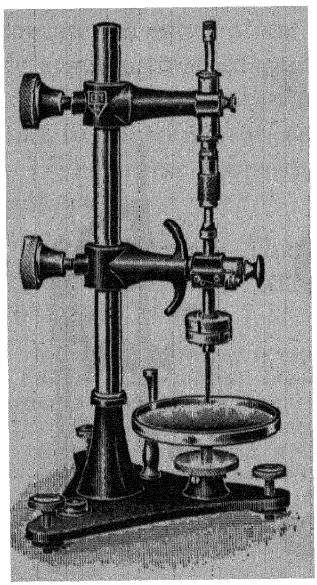


FIG. 221.—Penetrometer with Micrometer Attachment.

An ingenious automatic penetrometer has been devised by John Hutchinson [58] as illustrated in Fig. 222 (A), (B), (C), and (D), which operated in the following manner:

The timing mechanism is enclosed in a dust-proof "head" and the principle of operation is illustrated in the illustration of the "timing head" Fig. 222 (B) and the diagram Fig. 222 (C). Normally, the beam is locked in position by the spring loaded lever L which carries a friction pad bearing on the back of the beam. If required, during preliminary adjustment, the beam may be brought

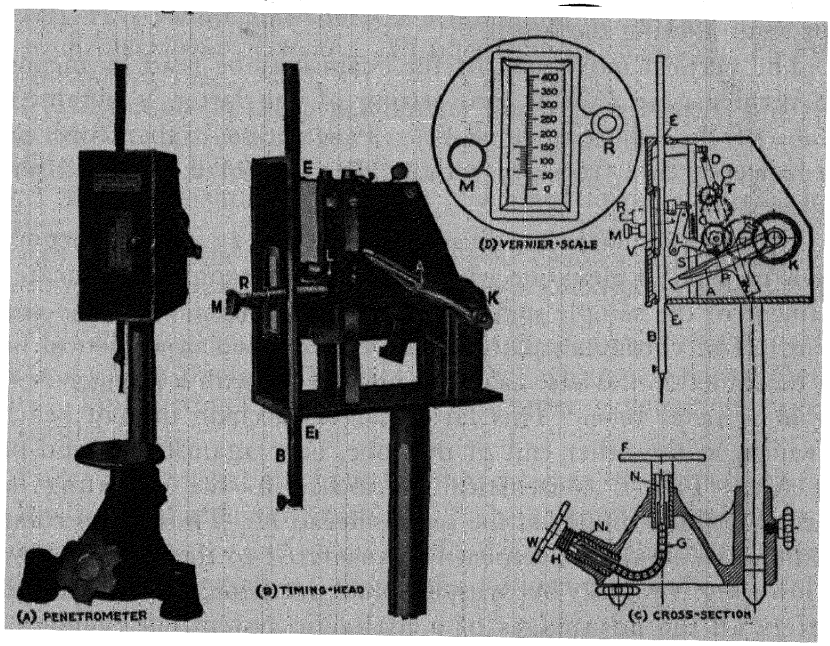


FIG. 222.—Hutchinson Automatic Penetrometer.

to a setting position by sliding it against this frictional lock. The other end of the lever L carries a short pin P which rides over the surface of a brass cam C. This latter is circular but has a slot S cut in the circumference into which the pin P may drop once per revolution.

When the pin is riding on the surface of the cam the brake lever L is held away from the penetrometer beam B and the latter is free to move vertically through the edge bearings E and E_1. Upon entry of the pin into slot S the lever L is allowed to move forward and lock the beam.

The cam is driven, through a chain of pinions, from a driving sprocket *K* which is impulsed by a short, stout driving spring *D*—the driving sprocket being loaded by depressing the external lever *A*. The cam wheel also drives, through a second train of pinions, a high speed escapement *T* which accurately controls the speed of rotation of the cam wheel. Upon releasing the beam by pressing the external trigger *R* the energy stored in the driving sprocket rotates the cam at a constant speed which is such that one revolution brings the slot into the engagement with pin *P* in exactly five seconds thus again locking the beam.

The vertical movement of the beam may be read by means of the vernier gear *V*. A zero setting of the latter is obtained by means of the adjusting knob *M*. Penetration is therefore easily read directly in 1/100 cm. without the need of a rack and pinion operated dial.

The sample platform, illustrated in Fig. 222 (C), incorporates a new method of elevation which allows very rapid but accurate adjustment of the sample surface to "scratch contact" with the needle point. The chromium plated table *F* is carried in a vertical bearing machined in the base casting, and is fitted with a plunger *N* sliding in a metal tube. This latter carries a chain of ball bearings *G* which, at the other end of the tube, bear against a second plunger *N*1. This is concentrically attached to the operating hand wheel *W* which rotates in the screw thread *H*. Thus, upon rotating the hand wheel the movement is transmitted to the table *F*, raising or lowering it rapidly but with absolute precision. This mechanism confers all the advantages of a hydraulic system but removes the possibility of leakage of oil or operating fluid. The inconvenience attached to the rotation of a screw-mounted table is also obviated but the platform is capable of rotation, if necessary, during final adjustment.

Assuming that the sample has been adjusted accurately to "scratch contact" with the needle, the vernier is set to zero relative to the graduations on the beam. The lever, situated on the right hand side of the head is then depressed to the full extent. Upon pressing the operating trigger, situated to the right of the vernier window, the beam will be released but automatically locked again after a five second period. The penetration may then be read di-

rectly from the relative position of the beam graduations and those on the vernier.

The principles underlying the vernier are very well known, but a diagram of the gear in this instrument is shown in Fig. 222(D). The vernier indicates a penetration of 64°.

(1) The instrument gives absolutely accurate timing of the penetration period thus eliminating possible errors due to manual operation with the aid of a stop watch. The high speed escapement controls the penetration period to an accuracy of 1/10 sec.

(2) It is robust and contains a minimum of moving parts subject to displacement or wear. All operating mechanism is enclosed in a dustproof case.

(3) Friction on the penetrometer beam is extremely small because the bearing surfaces are very short and well spaced. It is impossible for the beam to become jammed or to rock sideways—a defect which is common on some machines using a single, long bearing.

(4) The sample platform lifting gear enables rapid and precise adjustment to be made. The mechanism is enclosed in a heavy base casting and cannot jam or become unstable.

(5) The vernier system of reading penetrations eliminates the rack-and-pinion operated pointer and dial which is very subject to wear. The method gives direct reading in penetration degrees (1/100ths cm.).

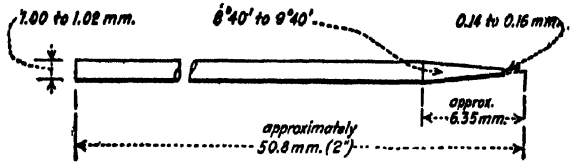
Careful investigations have been made as to the diameter of the holder for the bituminous material;[54] the method of preparing the specimen;[55] the size and shape of the needle;[56] also other variable factors.[57] As a result of these, the following standard test has been adopted:[58]

Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 g., 5 seconds, 25° C. (77° F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

The container for holding the material to be tested shall be a flat-bottom, cylindrical metal or glass dish, 55 mm. (2 9/16 in.) in diameter and 35 mm. (1 3/8 in.) deep. The needle (Fig. 223) for this test shall be made from a cylindrical steel rod approximately

50.8 mm. (2 in.) long, and having a diameter of 1.00 to 1.02 mm. This shall be symmetrically tapered at one end to a cone approximately 6.35 mm. (¼ in.) in height and whose angle shall be within the range of 8° 40′ and 9° 40′. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. The finished needle shall be hardened and highly polished.

The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be sup-



Courtesy A.S.T.M.

FIG. 223.—Needle for Penetration Test.

ported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath. Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. (⅝ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18° C. (65° F.) for one hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

In making the test, the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to completely cover the container. The transfer dish containing the

sample shall then be placed upon the stand of the penetration machine. The needle loaded with specified weight shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle-point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated.

At least three tests shall be made at points on the surface of the sample not less than 1 cm. (3/8 in.) from the side of the container and not less than 1 cm. (3/8 in.) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

When desirable to vary the temperature, time and weight, and in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested:

At 0° C. (32° F.) 200-g. weight, 60 seconds.
At 46.1° C. (115° F.) 50-g. weight, 5 seconds.

When three penetrations at three different temperatures, all ascertained with a given weight acting for a fixed time period, are known, it is possible accurately to plot the penetration-temperature curve of the particular specimen of bituminous substance,[59] by means of the following equation:

$$p = A + (BC^t)$$

where p = penetration in decimillimeters; t = the temperature in °C.; and A, B and C = constant characteristics of the particular substance under examination. If the three temperatures in °C. are so chosen that:

$$t_3 - 2t_2 + t_1 = 0$$

(e.g., 50°, 25°, and 0° C., or else 40°, 20° and 0° C.), where 0° C. is taken for t_1, then the first stated equation will assume the following simple form:

$$\frac{p_3 - p_2}{p_2 - p_1} = C^{t_2}$$

By plotting "log $(p - A)$" against "t", a *straight line* is obtained for any particular substance, from which it is possible to calculate or read off the constants B and C. The slope of the straight line represents "log C" and its intercept with the ordinate equals "log B." It should be noted that B is a dimensional constant, which by being suitably modified will enable any temperature scale to be employed.

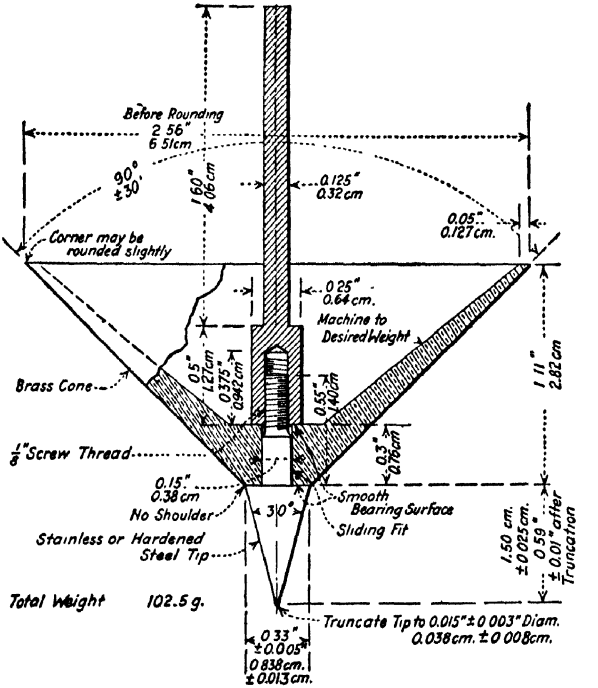
It has also been shown that if the time of penetration is maintained constant, there exists a fixed relation between weight and depth of penetration at varying temperatures. In all cases, the ratio of two penetrations obtained with two different weights remains practically constant over the entire temperature range. This fact may be utilized whenever it is desirable to measure penetration with weights either smaller or greater than 100 grams.

The following modification is used for testing the penetration of greases (i.e., the original and the worked consistency of No. O cup-grease and all harder greases) and of petrolatum:[60]

The method shall not be applied to greases which have been melted after having been poured and cooled during manufacture. Petrolatum, however, should be melted, poured into a suitable container and cooled before testing. In this test the needle is replaced by a cone shown in Fig. 224, which shall be constructed of stainless steel or of brass with a detachable hardened steel or stainless steel tip and made in accordance with the outside dimensions and tolerances shown. The interior construction and dimensions without tolerances are intended to be suggestive only. The outside surface of the cone and tip shall be given a very smooth finish. The total moving weight in the test shall be 150 g.* If the asphalt plunger weighs 47.5 g. (50 g. – 2.5 g. needle) the cone or the cone plus an attached weight must weigh 102.5 g. It is in general desir-

* The cone may be combined with the asphalt plunger if desired, providing the total weight of cone and plunger and the outside dimensions of the cone are not thereby changed.

able to make the cone as light as possible so that it will have a wide range of usefulness, and to add a weight to give the desired total for the grease test. If the conditions of the tests are not specified, the penetration of a grease shall be understood to refer to the results of this test made at 77° (25° C.).

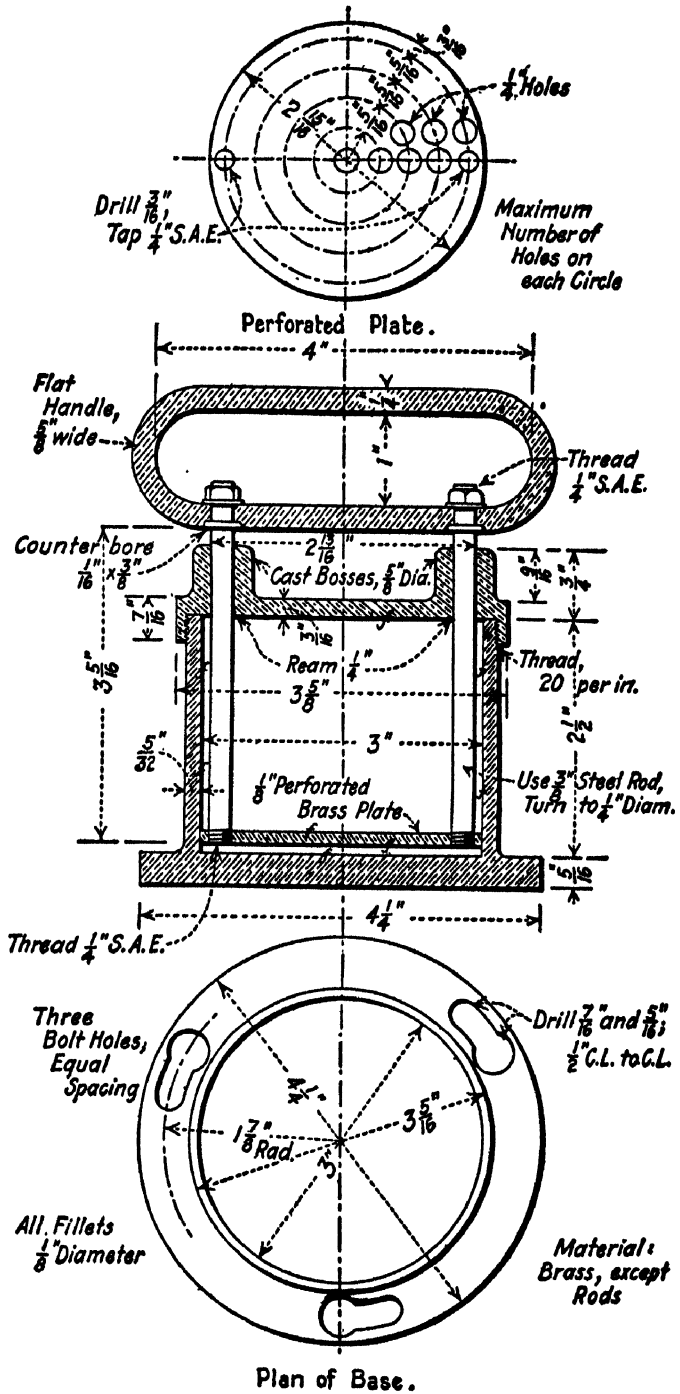


Courtesy A.S.T.M.

FIG. 224.—Penetrometer Cone.

The grease worker shown in Fig. 225 shall be used to work the grease to constant consistency in tests where the worked consistency is to be measured. A constant temperature bath regulated to 77° F. ± 1° F. (25° C. ± 0.5° C.) is desirable to bring the samples to the temperature of test if many tests are to be made.

Tests on the unworked consistency of grease shall be made only on grease in the original container (or cake in the case of very hard greases), 1-lb. tins being especially convenient for the purpose. If samples are to be taken from large containers, the tests must be made only on a sample worked as specified below. In taking samples from large containers, any discolored layers near the surface shall first be completely cut away and rejected. All samples of



Courtesy A.S.T.M.

FIG. 225.—Grease Worker.

petrolatum shall be tested for original (unworked) consistency after melting and cooling to the temperature of test.

(*A*) *Original* (*Unworked*) *Consistency.* The temperature of the sample shall be brought to within 1° F. (0.5° C.) of 77° F. (25° C.) before the test. If the sample is initially within 3 to 4° F. (1.5 to 2° C.) of this temperature, it can be brought to 77° F. (25° C.) by placing in a water bath for 30 or 40 minutes, but if the initial temperature is outside this range it must be placed in the constant temperature bath for 1½ hours to insure reaching the final constant temperature. If the room temperature is more than 3 to 4° F. (1.5 to 2° C.) from 77° F. (25° C.) a lid should be placed on the can, sealing with grease or petrolatum to prevent the entrance of water and the can immersed in the bath for the required period, as otherwise, the temperature of the surface will be different from that of the main body of the grease or petrolatum. Any holes or seams in the can should also be sealed with grease (petrolatum) or paraffin.

The surface of the grease or petrolatum shall be cut level and very smooth with a knife. Care must be taken not to work the surface of the grease or petrolatum. Any crust or discolored layers shall be completely removed before testing.

The can of grease or petrolatum shall be placed on the penetrometer table and the cone lowered until the tip just touches the surface. Watching the shadow of the tip aids in accurately setting the cone. The scale shall then be set to zero and the plunger released suddenly and kept released for five seconds. The penetration is read from the scale, the units being tenths of a millimeter.

In making tests, the total surface area disturbed by the test has a diameter about equal to the measured depth of penetration. In order to prevent one test from being affected by another disturbed area or by sides of the can, in starting a test the tip shall never be placed closer to the sides of the can or the edge of a previous hole than the penetration distance of that particular grease or petrolatum. The grease or petrolatum shall not be smoothed over for further tests.

Five tests shall be made on each sample and the average reported as the penetration, if the mean deviation of these readings does not exceed 3.0 per cent. If the mean deviation does exceed

3.0 per cent, the average of ten readings shall be reported as the penetration of the sample. More than one 1-lb. can will be needed for the required number of tests on the softer greases or petrolatums.

(*B*) *Procedure for Worked Consistency.* The procedure for worked greases shall be the same as that for unworked consistency except that the grease to be tested shall be transferred to the worker, which shall be filled heaping full with a minimum inclusion of air, brought to within 2° F. (1° C.) of 75° F. (24° C.) and worked with 60 double strokes of the plunger.* The top and plunger can then be removed, the surface smoothed over and the test made at once, if the temperature is within 1° F. (0.5° C.) of 77° F. (25° C.). As soon as one test is made, the surface may be smoothed over for the next, taking care to avoid creating air pockets. One 1-lb. sample is sufficient for all tests.

Various other methods have been proposed for testing the consistency of greases, including the measurement of the pressure required to force the substance through a capillary tube at a given temperature;[61] measuring the depth of penetration of a needle by means of a microscope;[62] etc. Similarly, a procedure has been suggested for measuring the consistency of asphalt putties and asphalt-fibrous roof coatings, based on the principles of a falling cylindrical rod and steel ball, respectively;[63] also a test measuring the extent of flow when subjected to pressure.[64]

The principal shortcoming of the needle penetrometer is the fact that the readings at various temperatures (115, 77 and 32° F. respectively) must be expressed on *different* scales, and are therefore not comparable. It is difficult, and in many cases impossible, to interpret the *extent* of the physical change from the range in readings, upon subjecting a bituminous substance to variations in temperature. In addition, the scope of the penetrometer is limited, as it will not answer for either semi-liquid or hard bituminous materials. These objections are overcome in the consistometer.

Test 9c. Consistometer. This instrument is constructed according to scientific principles, and may be accurately duplicated at any

* In general quite similar results may be obtained by working the grease thoroughly for five minutes with a spatula on a flat plate, providing care is taken to prevent working in much air. For referee tests, however, the standard worker must be used.

time. It registers the degrees hardness on a scale ranging from 0 to 100, and is suitable for determining the hardness of substances as soft as vaseline (which will test 0.3 at 77° F.) to substances as hard as gilsonite (testing in the neighborhood of 100 at 77° F.). In all cases, the hardness or consistency is expressed as the cube root of the number of grams which must be applied to a circular flat surface 1 sq. cm. (100 sq. mm.) in area, to cause it to displace the substance at a speed of 1 cm. per minute. Readings for all bituminous substances and at all temperatures (whether 115, 77 or 32° F.) are expressed on a *single* scale. The harder the substance, the greater will be its hardness expressed numerically.

Four mushroom-shaped plungers are used, each having a round flat head with a reduced shank, so the perimeter of the penetrating surface forms a "knife" edge. This entirely eliminates the frictional adhesion of the bituminous substance to the sides of the plungers. The flat heads of the plungers are made in the following dimensions:

Plunger	Diameter in mm.	Area in sq. mm.
No. 1	1.13	1
No. 10	3.57	10
No. 100	11.28	100
No. 1000	35.67	1000

The method of testing consists in forcing one of the plungers into the substance at a *uniform* speed of 1 cm. per minute. The force is automatically registered in grams or kilograms. For any plastic substance the number of grams required to effect this displacement is directly proportional to the *volume* displaced. The volumes displaced per minute by the respective plungers are 0.01, 0.10, 1.00 and 10.0 ml. respectively. The relation between the plungers is therefore in the direct proportion of 1 : 10 : 100 : 1000.

For the sake of uniformity, all readings are expressed in terms of the number of grams applied to plunger No. 100 (1 sq. cm.). In other words, the readings obtained with plunger No. 1000 are divided by 10, those obtained with plunger No. 10 are multiplied by 10, and those obtained with plunger No. 1 are multiplied by 100.

The hardness or consistency is equal to the cube root of this number of grams.

Two interchangeable springs are supplied, one registering in grams on a scale ranging 0 to 1000 g., in 10-g. divisions, and the other for reading in kilograms on a scale ranging from 0 to 10 kgs., in 0.1-kg. divisions. In using plungers No. 1 and No. 10, the kilogram spring *only* should be employed; in using plunger No. 100 either the gram or the kilogram spring may be employed, depending upon the hardness of the material; in using plunger No. 1000, the gram spring *only* should be employed. The relations are expressed in the following table:

TABLE XC

Plunger	Spring	Actual Reading	Converted to Grams per 100 sq. mm. Plunger	Cube Root Grams Applied 100 sq. mm. Plunger
1000 sq. mm.	G.	From 10 g. to 1000 g.	1 100	1.00 4.64
100 sq. mm.	G.	From 100 g. to 1000 g.	100 1,000	4.64 10.00
	Kg.	From 1.0 kg. to 10.0 kg.	1,000 10,000	10.0 21.5
10 sq. mm.	Kg.	From 1.0 kg. to 10.0 kg.	10,000 100,000	21.5 46.4
1 sq. mm.	Kg.	From 1.0 kg. to 10.0 kg.	100,000 1,000,000	46.4 100.0

Table XCI shows the relation between the consistometer readings and degrees hardness, bearing in mind that in every case the hardness is designated as the cube root of the number of grams applied to the No. 100 plunger (area 100 sq. mm.), to cause it to displace the substance at a speed of 1 cm. per minute.

The consistometer is illustrated in Fig. 226. It is first levelled by the four screws *A*. The spring *B* is then attached, selecting the gram spring for soft substances, or the kilogram spring for hard substances. The steel shaft *C* is inserted and screwed firmly into place. The plunger *D* should then be screwed into the lower end of the shaft. Plunger No. 1 is used for hard and brittle substances, plunger No. 10 for moderately hard solid substances, plunger No. 100 for moderately soft semi-solid substances, and plunger No. 1000 for semi-liquid substances.

TABLE XCI

For Converting Consistometer Readings into Degrees Hardness

Plunger No. 1000 (1000 sq. mm.)—Gram Spring

Grams Applied	0	10	20	30	40	50	60	70	80	90
0	0.00	1.00	1.26	1.44	1.59	1.71	1.82	1.91	2.00	2.08
100	2.15	2.22	2.29	2.35	2.41	2.47	2 52	2 57	2.62	2.67
200	2.71	2.76	2 80	2.84	2.88	2.92	2 96	3.00	3.04	3.07
300	3.11	3 14	3 17	3 21	3.24	3.27	3.30	3.33	3.36	3.39
400	3.42	3.45	3.48	3.50	3.53	3.56	3.58	3.61	3.63	3.66
500	3.68	3.71	3.73	3.76	3.78	3.80	3.83	3.85	3.87	3.89
600	3.91	3.94	3 96	3.98	4.00	4.02	4 04	4.06	4.08	4.10
700	4.12	4.14	4 16	4.18	4 20	4 22	4 24	4.25	4.27	4.29
800	4.31	4.33	4.34	4 36	4.38	4.40	4 41	4.43	4.45	4.46
900	4 48	4 50	4 51	4.53	4 55	4 56	4.58	4.59	4.61	4.63

Plunger No. 100 (100 sq. mm.)—Gram Spring

100	4.64	4 79	4.93	5 07	5 19	5.31	5 43	5.54	5.65	5.75
200	5.85	5.94	6.04	6 13	6.21	6.30	6.38	6.46	6.54	6.62
300	6.69	6 77	6.84	6 91	6.98	7.05	7.11	7.18	7.24	7.31
400	7 37	7.43	7 49	7 55	7.61	7.66	7 72	7.775	7.83	7.88
500	7.94	7.99	8.04	8 09	8 14	8.19	8 24	8.29	8.34	8.39
600	8.43	8.48	8.53	8.57	8.62	8.66	8.71	8.75	8.79	8.84
700	8.88	8.92	8 96	9 00	9 045	9.09	9.13	9.17	9.21	9.24
800	9.28	9 32	9.36	9.40	9 44	9 47	9.51	9.55	9.58	9.62
900	9.65	9.69	9 73	9 76	9 80	9 83	9 86	9.90	9.93	9.97

Plunger No. 100 (100 sq mm.)—Kilo Spring

Kilos Applied	0.0	0.1	0.2	0.3	0.4	0 5	0 6	0.7	0.8	0.9
1.0	10 0	10 3	10 6	10 9	11 2	11.4	11 7	11 9	12 2	12.4
2.0	12.6	12 8	13 0	13 2	13.4	13.6	13.75	13.9	14.1	14.3
3.0	14.4	14.6	14.7	14.9	15 0	15 2	15 3	15.5	15.6	15.7
4.0	15.9	16 0	16.1	16.3	16.4	16.5	16.6	16.75	16.9	17.0
5.0	17.1	17 2	17 3	17 4	17.5	17.65	17.8	17.9	18.0	18.1
6.0	18.2	18 3	18 4	18.5	18.6	18.7	18.8	18.85	18.95	19.0
7.0	19.1	19.2	19.3	19.4	19 5	19.6	19.7	19.75	19.8	19.9
8.0	20.0	20 1	20.2	20.25	20.3	20.4	20.5	20.6	20.65	20.7
9 0	20 8	20 9	20 95	21 0	21.1	21.2	21.25	21.3	21.4	21.5

Plunger No. 10 (10 sq. mm.)—Kilo Spring

1.0	21.5	22.2	22.9	23.5	24.1	24.7	25.2	25.7	26.2	26.7
2.0	27.1	27.6	28.0	28.4	28.8	29.2	29.6	30.0	30.4	30.7
3.0	31.1	31.4	31.7	32.1	32.4	32.7	33.0	33.3	33.6	33.9
4.0	34 2	34.5	34.8	35.0	35.3	35 6	35.8	36.1	36.3	36.6
5.0	36.8	37.1	37.3	37.6	37.8	38.0	38.3	38.5	38.7	38 9
6.0	39 1	39 4	39 6	39.8	40.0	40.2	40.4	40.6	40.8	41.0
7.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.5	42.7	42.9
8.0	43.1	43 3	43 4	43.6	43.8	44.0	44.1	44.3	44.5	44.6
9.0	44 8	45 0	45 1	45 3	45 5	45 6	45.8	45.9	46.1	46.3

Plunger No. 1 (1 sq. mm.)—Kilo Spring

1 0	46.4	47.9	49.3	50 7	51 9	53.1	54 3	55.4	56.5	57.5
2.0	58.5	59.4	60.4	61.3	62 1	63.0	63.8	64.6	65.4	66.2
3.0	66.9	67.7	68.4	69.1	69.8	70.5	71.1	71.8	72.4	73.1
4.0	73 7	74.3	74.9	75.5	76.1	76.6	77.2	77.75	78.3	78.8
5.0	79.4	79.9	80.4	80.9	81.4	81.9	82.4	82.9	83.4	83.9
6.0	84.3	84.8	85.3	85.7	86.2	86.6	87.1	87.5	87.9	88.4
7.0	88.8	89.2	89.6	90.0	90.45	90.9	91.3	91.7	92.1	92.4
8.0	92.8	93.2	93.6	94.0	94.4	94.7	95.1	95.5	95.8	96.2
9.0	96.5	96.9	97.3	97.6	98.0	98.3	98.6	99.0	99.3	99.7
10.0	100.0	100.3	100.7	101.0	101.3	101.6	102.0	102.3	102.6	102.9

The scale E is graduated in grams on one side, and kilograms on the other, and is reversible. It should be inserted so that the graduations will correspond with the spring used, and adjusted so the indicator F will rest at the o division. The maximum indicator G is also brought to the o division, using the small instrument H.

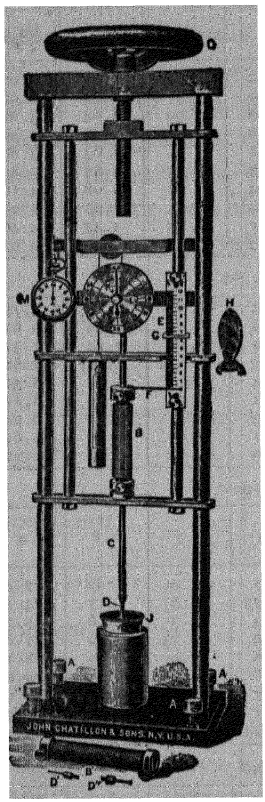


FIG. 226.—Consistometer.

The bituminous substance is melted at the lowest possible temperature and poured into a small receptacle as described for the needle penetration method. The tin box J containing the bituminous substance is then supported underneath the machine in a vessel of water (not shown) maintained at the temperature at which the test is to be performed. The pressure is applied to the plunger by turning the hand-wheel O, and the speed of displacement controlled by following the pointer K, on the dial L, which should be caused to revolve at the same speed as the second hand of a chronometer M, conveniently suspended alongside. The numbers on the dial L correspond with those of the second hand on the chronometer. One revolution of the pointer K indicates that the plunger has moved downward exactly 1 cm.

At the termination of sixty seconds, after the pointer on the dial has made one revolution, the pressure on the plunger is relieved. The reading of the maximum indicator G on the scale E is then noted, and the corresponding degree of hardness ascertained by referring to the table.

When the plunger commences to displace the substance at the specified speed of 1 cm. per minute, a maximum reading is obtained which should remain constant throughout the entire displacement. The consistometer is simple to operate, gives closely concordant results, expresses the readings obtained at all temperatures on *one scale* and has a sufficiently great range to include all bituminous substances ordinarily encountered.[65]

Test 9d. Susceptibility Index. Various methods have been suggested for evaluating the susceptibility of bituminous substances, so as to indicate the change of consistency or hardness with changes in temperature. It has been observed that substances having low susceptibility to temperature changes possess a high degree of plasticity, elasticity and thixotropy.[66]

The Saybolt-Furol viscosimeter (Test 8*b*) may be used for measuring the susceptibility of bituminous substances having viscosities between 1 and 5 poises (approximately 50 to 250 Furol seconds) either at room temperatures, or in the melted state at "processing" temperatures, so as to indicate the percentage change of viscosity for each degree rise in temperature.[67] Similarly, the alternating stress method (Test 8*g*) or the falling coaxial cylinder method (Test 8*h*) may be employed for measuring the susceptibility of bituminous substances in terms of their viscosities within the temperature range of approximately 60 to 100° F. (i.e., at their so-called "service" temperature).[68] In these regions, the log viscosity-temperature chart is a straight line, within the limits of experimental error, and the following relationship prevails:

$$\log \eta = at + \log b$$

where η = viscosity in poises; t = temperature in °C.; and a also b are constants. The percentage change of viscosity is a function of the slope "a," because:

$$a = \frac{\log \eta_2 - \log \eta_1}{t_2 - t_1} = \frac{\log \eta_2/\eta_1}{t_2 - t_1}$$

where η_1 and η_2 are the viscosities at temperatures t_1 and t_2, respectively, consequently:

$$\text{Percentage change} = 100(10^{a(t_2 - t_1)} - 1)$$

The susceptibility may therefore be measured in terms of a viscosity index, which represents the percentage decrease in viscosity for 1 deg. C. rise in temperature. Experimentally, it is only necessary to measure the viscosities η_a and η_b at any two temperatures t_a and t_b respectively, whereupon the susceptibility index may be expressed as:

$$\text{Susceptibility Index} = 100\,(10^a - 1) = 100\,[\eta_a/\eta_b)^{1/(t_a - t_b)} - 1]$$

Furol viscosities are converted into absolute units (poises) by means of the following formula:

$$\frac{\eta}{d} = 0.0216\,F$$

where η = viscosity in poises; d = density in grams per ml.; and F = Furol seconds.

The author has observed that in the case of bituminous substances derived from a similar source, and regardless of their consistency or fusing-point, the range in consistometer hardness between 115 and 32° F. divided by the K. and S. fusing-point in deg. F. is equal to a "constant." This constant has been termed the "susceptibility index" of the bituminous substance. The more susceptible the material is to temperature changes, the higher will be its "susceptibility index." It is calculated from the consistometer hardness and the K. and S. fusing-point (Test 15*a*) in the following manner:

$$\text{Susceptibility Index} = \frac{(Hd \text{ at } 32^\circ \text{ F.}) - (Hd \text{ at } 115^\circ \text{ F.})}{\text{Fusing-point, K. and S. Method}} \times 100$$

This index is useful as a means of identification; for predetermining the adaptability of the substance for certain usages; gauging its uniformity of supply; and for purposes of factory control. This index bears no relation to the hardness or fusing-point, and is substantially constant for bituminous substances derived from the same source or produced by the same general process. In the case of blown petroleum asphalts, it furnishes an indication of the *extent* to which the substance has been blown; the further the process having been continued, the smaller will be the index expressed numerically. With residual asphalts derived from any one crude, the susceptibility index will remain constant, regardless of the extent to which the distillation has progressed.

By means of the susceptibility index, bituminous materials may be roughly divided into the following groups, viz.:

Susceptibility Index under 40: Includes blown petroleum asphalts, fatty-acid pitches and fluxed asphaltites (having an index between 8 and 40); also wurtzilite asphalts (having an index between 30 and 40).

Susceptibility Index between 40 *and* 60: Includes residual asphalts.

Susceptibility Index over 60: Includes mineral waxes, pitches derived from tars, and asphaltites (of which the susceptibility index varies from 75 to over 100).

Native asphalts have been excluded from the foregoing groups, since their susceptibility indices vary widely, ranging from 15 to greater than 100. The author has never examined a bituminous material having a susceptibility index lower than 8.[69]

Other formulas have been proposed from time to time for expressing the susceptibility of bituminous substances,[70] including the following:

$$\text{Susceptibility index} = \frac{\text{penetration at } 46.1^\circ \text{ C. per 50 grams per 5 seconds} - \text{penetration at } 0^\circ \text{ C. per 200 grams per 60 seconds}}{\text{penetration at } 25^\circ \text{ C. per 100 grams per 5 seconds}}$$

$$\text{Susceptibility index} = \frac{\text{penetration at } 37.8^\circ \text{ C. per 100 grams per 5 seconds}}{\text{penetration at } 25^\circ \text{ C. per 100 grams per 5 seconds}}$$

$$\text{Susceptibility index} = \frac{\text{penetration at } 25^\circ \text{ C. per 100 grams per 5 seconds}}{\text{penetration at } 0^\circ \text{ C. per 200 grams per 60 seconds}}$$

DUCTILITY

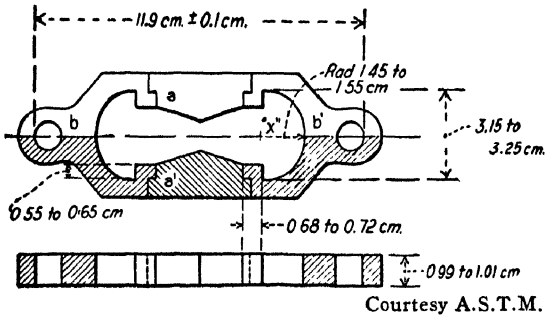
This represents the capacity of the bituminous material for elongating or stretching. The test is of value for identifying the bituminous substance, for indicating its adaptability in connection with certain usages, for gauging its uniformity of supply, and for purposes of factory control. The ductility test will often differentiate blown petroleum asphalts from native or residual asphalts. Most pitches derived from tars are extremely ductile, but fatty-acid pitches are variable in this respect. The ductility test is useful for predetermining the adaptability of bituminous materials for paving purposes, for adhesive compounds to be used in connection with waterproofing or built-up roofing work, and for manufacturing surface coatings of preparing roofings. Wherever the bituminous material is subjected to extensive changes in temperature or vibration, it is important that it should have high ductility within the particular temperature range to which it will be subjected. With every bituminous substance there exists a certain temperature, usually within 10 to 30° F. of its fusing-point (K. and S. method), at which the ductility attains a maximum. A ductility curve constructed for any bituminous substance over a range of temperature assumes the same form as the probability curve in higher mathematics. It is desirable

that the maximum ductility should coincide as closely as possible with the *average* temperature to which the material is to be subjected.

There are two methods in use, depending upon the construction of the molds, namely one devised by A. W. Dow,[71] and one proposed by the author.

Test 10a. Dow's Method. This test has been standardized as follows:[72]

The ductility of a bituminous material is measured by the distance to which it will elongate before breaking when two ends of a briquet of the material of the form described are pulled apart at a specified speed and at a specified temperature. Unless otherwise



Courtesy A.S.T.M.

FIG. 227.—Ductility Mold.

NOTE.—The opening in the end of each clip, as indicated by "x," shall be half an ellipse having a transverse axis of 3.2 cm. ± 0.05 cm. and half of the longitudinal axis shall be 1.45 to 1.55 cm.

specified, the test shall be made at a temperature of 25° C. ± 0.5° C. (77° F. ± 0.9° F) and with a speed of 5 cm. per minute (± 5.0 per cent).

The mold shall be similar in design to that shown in Fig. 227. Dimensions shall be as given with the permissible variations indicated. The mold shall be made of brass, the ends, *b* and *b'*, being known as clips, and the parts, *a* and *a'*, as sides of the mold. The dimensions of the mold shall be such that, when properly assembled, it will form a briquet having the following dimensions:

Total length	7.45 to 7.55 cm.
Distance between clips	2.97 to 3.03 cm.
Width at mouth of clip	1.98 to 2.02 cm.
Width at minimum cross-section (halfway between clips)	0.99 to 1.01 cm.
Thickness throughout	0.99 to 1.01 cm.

The water bath shall be maintained at the specified test temperature varying not more than 0.1° C. (0.18° F.) from this temperature. The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. and shall be supported on a perforated shelf not less than 5 cm. from the bottom of the bath.

For pulling the briquet of bituminous material apart, any apparatus may be used that is so constructed that the briquet will be continuously immersed in water as specified, while the two clips are pulled apart at a uniform speed, as specified, without undue vibration.

The bituminous material to be tested shall be completely melted until thoroughly fluid by heating it in an oil bath maintained at the minimum temperature needed to properly liquefy the sample (see Note). It shall then be strained through a No. 50 sieve and, after a thorough stirring, poured into the mold. The mold shall be assembled on a brass plate and, so as to prevent the material under test from sticking, the surface of the plate and interior surfaces of the sides *aa'* of the mold shall be thoroughly amalgamated.* The plate upon which the mold is placed shall be perfectly flat and level so that the bottom surface of the mold will touch it throughout.[73] In filling the mold, care shall be taken not to disarrange the parts and thus distort the briquet. In filling, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall be left to cool to room temperature and then placed in the water bath maintained at the specified temperature of test for 30 minutes, after which the excess bitumen shall be cut off by means of a hot straight-edged putty knife or spatula so that the mold shall be just level full.

NOTE.—When paving asphalt cements are being tested, the oil bath shall be maintained at a temperature of from 125 to 150° C. (257 to 302° F.).

The brass plate and mold, with briquet, shall then be placed in the water bath and kept at the specified temperature for at least 1½ hours, when the briquet shall be removed from the plate, the side

* The amalgamation may best be effected by immersing the clean mold in a solution of mercury bisulphate containing free metallic mercury, and so as to come in contact with the latter. Instead of mercury, the metal mold, preferably of stainless steel, may be moistened with glycerol.[74]

pieces detached, and the briquet immediately tested. The rings at each end of the clips shall be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform speed as specified [75] until the briquet ruptures. A variation of ± 5 per cent from the speed specified will be allowed. The distance through which the clips have been pulled to produce rupture shall then be measured in centimeters. While the test is being made, the water in the tank of the ductility machine shall cover the sample both above and below it by at least 2.5 cm. and shall be kept continuously at the temperature specified within ± 0.5° C. (± 0.9° F.).

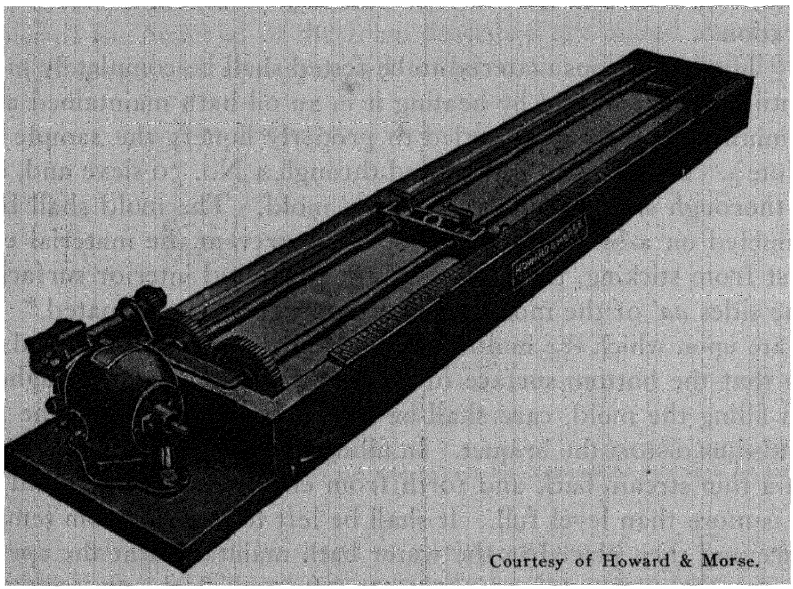


FIG. 228.—Smith Ductility Machine.

A normal test is one in which the material between the two clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross-sectional area. The average of three normal tests shall be taken and reported as the ductility of the sample.

If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal.

NOTE.—When the specific gravity of the bituminous material to be tested is below 0.98 or above 1.01, the specific gravity of the water bath in the ductility machine shall

be made the same as the material to be tested by the addition of either methyl alcohol or sodium chloride.

If a normal test is not obtainable on three successive tests, the ductility shall be reported as being unobtainable under the conditions of the test.

It is customary to find the ductility at three temperatures, viz.: 115°, 77° and 32° F. However, considerable insight may be obtained regarding the origin and characteristics of bituminous substances, by ascertaining the ductility over an extended temperature range, as may be noted from the following figures:[76]

TABLE XCII

DUCTILITY AT VARYING TEMPERATURES

Temp.	*A*	*B*	*C*	*D*	*E*	*F*	*G*	*H*
−3° C.	.....	.....	.....	.....	.....	.....	7.7	7.1
−2°	.....	.....	.....	.....	.....	.....	8.5	9.4
0°	.....	.....	.....	.....	0	.....	12	10.5
+1°	.....	.....	.....	.....	1.5	0		
1.5°	.....	.....	0	.....	.....	2 0		
2°	.....	.....	2	0	39.5	5	16	14.5
2.5°	.....	.....	.....	0.5				
3°	0	.....	8.2	.....	61	32.5		
3.5°	1							
4°	>100	.....	14	8	94	64	25	25
5°	.....	0	33	22	>100	>100	.....	30.5
6°	.....	1	61.5	32	.....	.....	34	
6.5°		>100						
7°	.....	.....	81	.....	.....	.....	41	46
7.5°	.....	.....	.....	69				
8°	.....	.....	>100					
9°	.....	.....	.....	98	.....	.....	52	
11°	.....	.....	.....	.....	.....	.....	65	79.5
13°	.....	.....	.....	.....	.....	.....	80.5	>100
15°	.....	.....	.....	.....	.....	.....	>100	.....

Where *A* represents soft coal-tar pitch.
B the same distilled to a fusing-point of 28° C. (loss 8.4 per cent).
C a mixture of coal-tar pitch and anthracene oil (70/30).
D the same as *C* distilled to a fusing-point of 28° C. (loss 9.0 per cent)
E a mixture of coal-tar pitch and heavy oil (75/25).
F the same as *E* distilled to a fusing-point of 28° C. (loss 9.5 per cent).
G and *H* soft residual asphalts having fusing-points of 28° C.

Various machines have been devised for ascertaining the ductility, in which the molds are drawn apart mechanically, including the one devised by Smith, illustrated in Fig. 228.[77]

An instrument with a dynamometer attachment adapted to use the Dow mold, measuring both the ductility and "cementitiousness" (tensile strength) has been described by Lester Kirschbraun.[78] This device is essentially the same as that which had been previously described by the author in 1910. (See Test 10*b*.)

It has been shown that partially blown asphalts may be prepared which will show a ductility at 32° F. of at least 1 cm. by the above method of testing, some samples running as high as 2.5 cm.[79]

Test 10b. Author's Method. An improved mold designed by the author, is illustrated in Fig. 229 and shown in cross-section in Fig. 230. It consists of two cylindrical sections constructed of hardened steel, resting together on circular knife-edges and maintained in that position by three guide pins. It is filled by unscrewing the upper cap and pouring in the melted bituminous substance, which on cooling forms a prismoid, whose altitude is 2.5 cm., the end-areas 1.8 cm. in diameter, with a minimum cross-section at the center of exactly 1.0 sq. cm. (1.28 cm. in diameter). The upper cap is screwed in place, the mold fastened in the tensometer and the two halves separated at the uniform speed of 5 cm. per minute. The elongation in cms. at the moment the material parts is a measure of its ductility.[80]

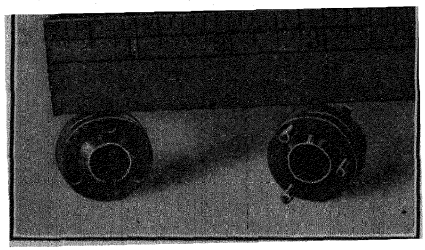


FIG. 229.—Author's Ductility Mold.

This mold has a number of advantages over the Dow type, including its adaptability to testing semi-liquid and semi-solid bituminous materials, no amalgamation is necessary, there is no danger of the material breaking in the mold upon being cooled to the proper temperature, the personal equation is eliminated in filling the mold with the assurance that the minimum cross-section will be *exactly* the proper size, and only a small quantity of the material is required in making the test.

The tensometer is illustrated in Fig. 231. The two sections of the mold *A*-1 and *A*-2 are clamped between the guides *B*-1 and *B*-2, the lower section being fastened to the stationary cross-piece *C* by the pin *D*-1, and the upper section to the movable cross-head *E* by the pin *D*-2. The cross-head is attached to the chain *F* which

passes over the sprocket-wheel G fastened to the dynamometer H, and then around a suitable winding mechanism I. The specimen is drawn apart by turning the handwheel J, which operates the endless chain K running on the sprocket wheels L-1 and L-2. The dynamometer is equipped with a trigger M to prevent recoil. The chain F also connects with a train of gears operating the brass pointer N pressing against the dial O which is formed of vulcanite or some other insulating material. Its face is marked in one hundred divisions, each consisting of a metallic contact. As the pointer brushes over these contacts it momentarily closes an electric circuit which

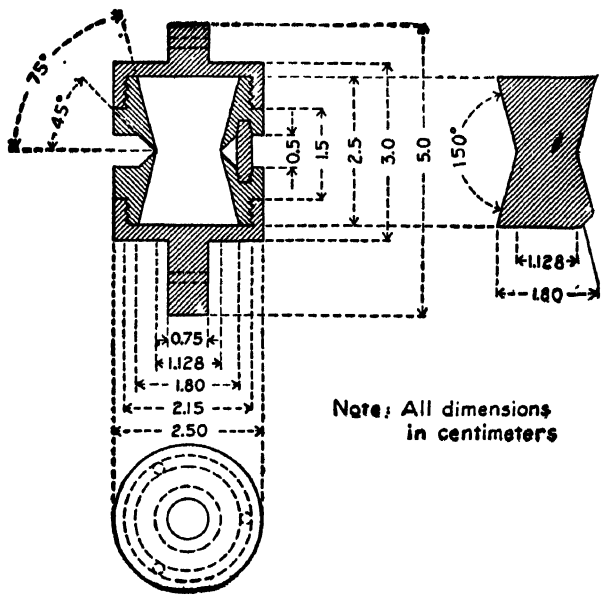


FIG. 230.—Cross-section, Author's Mold.

operates the relay P, causing a "click." The relay is connected with the batteries Q and the switch R. One revolution of the pointer indicates that the halves of the mold have been separated a distance of exactly one meter, and a movement of the pointer over one division of the dial corresponds to a centimeter rise in the section A-2.

The guides B-1 and B-2 are pivoted at S-1 and S-2, which permits the glass reservoir T being slipped into place, whereupon they are locked into position by the bolt U. The reservoir is filled with water maintained at the desired temperature by the heating coil Y in series with the incandescent lamps V. The bath may be agitated

upon squeezing the bulb *W*, which forces air through the liquid. The valve *X* is for emptying the reservoir. The speed is controlled by a metronome with a bell attachment set to ring every 12 seconds, or 5 times per minute. The speed with which the crank is turned must be regulated so that the "clicks" of the relay are brought into unison with the rings of the metronome.

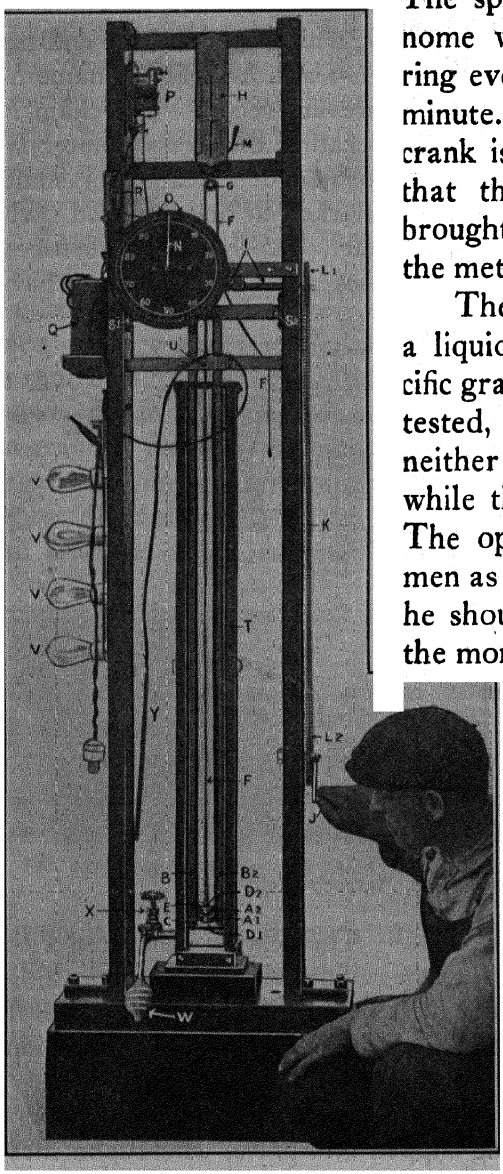


FIG. 231.—Tensiometer.

The reservoir should be filled with a liquid having about the same specific gravity as the bituminous material tested, so the thread of material will neither have tendency to float nor sink while the molds are being separated. The operator must watch the specimen as sections *A* and *B* separate, and he should cease turning the crank at the moment the thread parts. The dynamometer indicates the tensile strength of the substance in kilograms (Test 11), and the dial *O* records its ductility in centimeters. The substance is usually tested at 115, 77 and 32° F.

TENSILE STRENGTH (COHESIVENESS)

Cohesion (cohesiveness) is defined as the force by which molecules of the same substance are held together, and causes the substance to resist being pulled apart. The cohesion of bituminous substances is a function of the hardness (penetration). The softer

the substance (derived from the same source), the less will be its cohesiveness. The cohesion of a liquid at a given temperature has been shown to be equal to twice its surface tension at the same temperature. This may be explained by the fact that molecules of a liquid are surrounded by a field of force, and when the molecules are in the body of the liquid, this attraction for its neighbors is the same in all directions. However, the molecules at the boundary between the liquid and air are attracted to one another laterally on the surface only, creating a tension, known as the surface-tension.

Test 11. Author's Method. The tensile strength (cohesiveness) of bituminous substances may be measured on the tensometer as described in Test 10*b*, and is equal to the maximum reading in kilograms as the two halves of the mold separate. It is a measure of cohesiveness or cementitiousness and is of value in determining the adaptability of a bituminous substance for certain definite purposes, especially for paving, manufacturing adhesive compounds for waterproofing and built-up roofing work, bituminous substances for electrical insulation, molded articles, pipe joints, etc. The tensile strength is ordinarily tested at 115, 77 and 32° F. For each bituminous substance, there is a critical temperature at which the tensile strength reaches a maximum, and this is generally coincident with the temperature at which the ductility approaches 0. This phenomenon may be explained by the disappearance of plasticity and associated cohesiveness at temperatures when the substance becomes transformed into a brittle solid. The tensile strength curve is also similar in form to the probability curve in higher mathematics. There appears to be no definite relation between the hardness and tensile strength of bituminous substances. With residual asphalts manufactured from the same crude, the tensile strength is reduced after the distillation progresses beyond the hard and brittle stage. Excessive blowing produces the same results, but to a lesser degree.

ADHESIVENESS

Adhesion (adhesiveness) is defined as the force by which one substance adheres to another of a different nature.[81] This test serves as a measure of the adhesiveness of the bituminous material, and it is of primary importance in ascertaining its adaptability for certain definite usages, as, for example, in road building, preparing

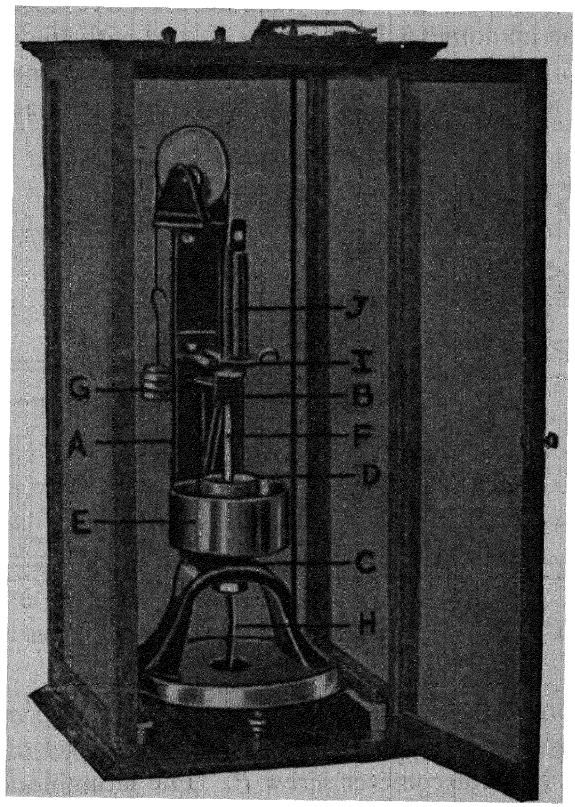
compounds for waterproofing and built-up roofing work, cements, etc. It represents the capacity of the substance to adhere to solid objects with which it may be brought in contact, and differs entirely from the cohesiveness or tensile strength referred to in Test 11. Various instruments have been proposed for this purpose, including those devised by Fulweiler,[82] Osborne,[83] Brown, Kirschbraun and others.[84]

Test 12a. Riehm's Method. This test constituted a rough indication of the adhesiveness of the substance and is ascertained by noting the number of seconds time it takes for a 10-gram weight (the underside of which is smooth and clean) to adhere to a freshly prepared surface of the bituminous substance at 20° C.[85]

Test 12b. Wedmore's Method. A cylindrical steel mold, open at both ends, is allowed to rest against the surface of a polished steel plate, and filled with the melted bituminous substance at a predetermined temperature. Upon cooling to 77° F., the mold is subjected to a steady, slow pull, and the force measured which is necessary to separate the mold from the plate. The area of adhesion should be $\lessdot$ 90 per cent of the whole area covered by the specimen.[86]

Test 12c. Brown's Method. Performed by means of an instrument designed by W. A. Brown, termed the "adhesivemeter," for measuring the stickiness or cohesive strength of road oils. The instrument is illustrated in Fig. 232 and consists of a cast-iron frame *A* carrying two brass bearings. The upper bearing *B* is 2 in. long and the lower bearing *C* is ½ in. in diameter and ½ in. long, attached to an oil-cup *D* which in turn is surrounded by the water bath *E*. A steel rod *F* passes through the bearings and is counterbalanced by the weight *G*. The testing weights are suspended from the wire *H* attached to the lower end of the rod. A clamp *I* holds the rod in position prior to the test. In running the test, the oil-cup *D* is filled with the road oil which serves to coat the rod before it reaches the lower bearing *C*. When the oil has reached the desired temperature, controlled by the water bath provided with an electric heater and thermostat, the rod is raised until its upper end reaches the zero mark on the scale *J*. The clamp is then released, and when the upper end of the rod has fallen to the 3-cm. mark, a stop-watch is started and timed until it reaches the 13-cm. mark.

The time in seconds for the rod to fall 10 cm. is an index of the adhesiveness of the material. The test is usually run at 95° F. with a weight of 760 g. suspended from the lower end of the rod.



Courtesy of Braun Corp.

FIG. 232.—Brown Adhesivemeter.

SURFACE TENSION

Test 12d. Nellensteyn's Method. A special form of apparatus has been devised by F. J. Nellensteyn [87] for ascertaining the surface-tension of bituminous substances, as shown in Fig. 233, consisting of a manometer which is formed of two hollow cylinders *A* and *B* joined together by an air-tight closed coupling *C*. The cylinders are partly filled with dekalin (*D*) which supports a duraluminium float *E* of accurate dimensions. This float is attached to a weighing device. As the pressure in the gas-chamber of the manometer

increases, the pointer F moves upwards and can be recorded by the weight G. Purified nitrogen gas (washed with alkaline pyrogallol, followed by alkaline manganese chloride solution) enters at H and the pressure is regulated by the needle-valve J, which is connected with the manometer by the tube K, and with the capillary M by the tube L. The capillary is composed of a platinum-rhodium alloy and has a diameter at the tip of 0.6 mm. It is adjusted in a

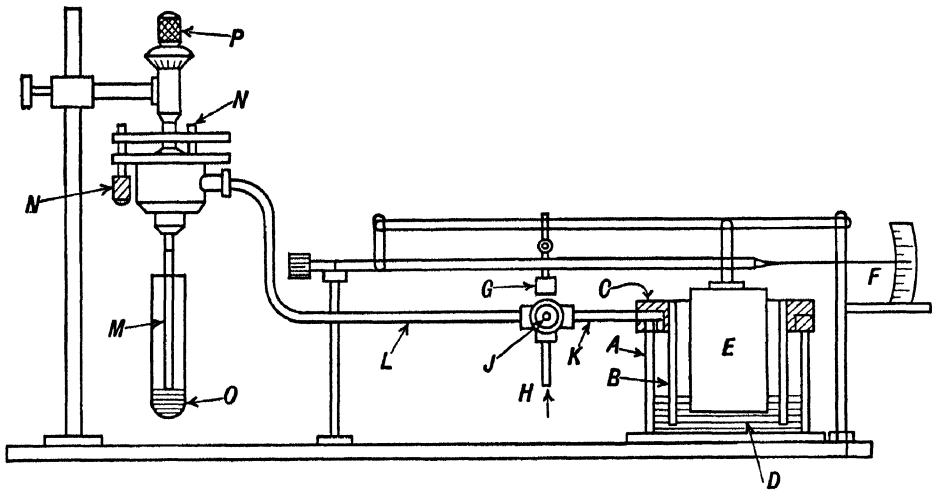


FIG. 233.—Apparatus for Determining Surface-Tension.

vertical position by the set-screws N, whereupon the tip of the capillary is introduced just below (i.e., 0.1 mm.) the level of the liquid to be tested O, by a micrometer screw P. The bituminous substance is maintained at the desired test-temperature by being surrounded by a bath of castor oil (not shown).

The apparatus is first calibrated against C.P. benzol, which has a surface-tension of 28.5 dynes/cm. at 20° C., with a temperature correction of 0.16 dyne/cm. per degree C. Upon making the test, the zero-point is obtained by allowing the gas to enter under the greatest pressure which will not, however, cause it to bubble through the substance under test. The pressure is then gradually increased until the gas bubbles through the substance at the rate of 1 bubble per minute, and thereupon gradually reduced until a point is reached where there is no noticeable further change in pressure. This ordinarily takes place when the bubbles form at the speed of one in

each 30 to 50 seconds, before they burst. This pressure is then recorded.

The surface-tension may be calculated by means of Cantor's equation, which in simplified form, gives the following:

$$x = \frac{rH}{2}$$

where x = surface-tension energy expressed in ergs per cm.^{2}; r = radius of the tip of capillary tube in mm.; H = pressure recorded in dynes (i.e., mm. mercury corrected to 0° C. × 1333.2 dynes).

The molecular surface-tension is calculated from the following formula:

$$\mu = x\left(\frac{M}{d}\right)^{\frac{2}{3}}$$

where μ = molecular surface-tension energy in ergs per cm.^{2}; M = molecular weight of the substance; d = specific gravity of the substance at the temperature of test, compared with water at 4° C.

Nellensteyn reports the figures given in Table XCIII, upon examining the following substances:

A. Blown Venezuelan petroleum asphalt (R. and B. fusing-point 64.2° C.).

B. Residual Mexican petroleum asphalt (R. and B. fusing-point 42° C.).

C. Residual Mexican petroleum asphalt (R. and B. fusing-point 55.7° C.).

D. Residual Mexican petroleum asphalt (R. and B. fusing-point 58.5° C.).

E. Trinidad asphalt separated from its associated mineral constituents by dissolving in CS_2 and centrifuging at 40,000 r.p.m. (R. and B. fusing-point 65° C.).

F. Coal-tar pitch obtained by distilling coal tar to 300° C. (R. and B. fusing-point 60.2° C., free carbon 10.7 per cent).

These investigations show that whereas all true liquids exhibit a changing surface-tension proportional to the temperature of test, both asphalts and pitches show a steadily falling surface-tension to a certain value, as the temperature rises, at which point the curve suddenly flattens, after which the surface-tension again becomes proportional to the temperature, the fall thereupon being more gradual than previously. This indicates a change of state at this

critical point, which has a different value for the different substances examined.

TABLE XCIII

SURFACE-TENSION OF ASPHALTS AND THEIR COMPONENTS

	A		B		C		D		E		F	
	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.
Surface Tension (S.T.) Original Substance:												
Range examined..... From...	117	33.2	93.5	31 9	117	31 9	116	32.7	108	37.4	67	39.7
To.....	225	20.7	179	23 8	224	22.1	215	22 3	193 5	25.8	180	33.8
Bend in S.T.-Temp. chart occurs at	150	24.8	117	27 5	145	26 7	138	26 8	131 5	30.1	80	38.8
d(S.T.) ÷ d (Temp.)* from bend in chart to highest temperature examined.....	0.0546		0.5095		0 0562		0 0584		0 0693		0.0530	
Surface Tension (S.T.) Separated Oily Constituents (Note A):												
Range examined..... From...	30	36 6	39 5	34 7	52	35 7	45	33 5	65	34.8	46	46.5
To.....	226	20 5	184 5	22 8	233	21 4	220	21 5	228	20.9	211	30.8
Bend in S.T.-Temp. chart occurs at	50	30.6	58	30 6	110	28 2	69 5	30 3	80	30.0	65	38.6
d(S.T.) ÷ d (Temp.)* from bend in chart to highest temperature examined.....	0.0575		0.0600		0.0553		0 0584		0 0615		0.0522	
Relation between S.T. Original Substance and Separated Oily Constituents (Note A).....												
Difference between S.T.s at 180° C. of original substance and separated oily constituents.....	—	0.25	—	0.60	—	0 35	—	0 50	—	2.85	—	1.25
Difference between temperatures of Bends in S.T.-Temp. charts of original substances and separated Oily Constituents.....	100	—	65	—	65	—	70	—	55	—	15	—

* The temperature coefficient of surface-tension per °C.

NOTE A.—The separated oily constituents (i.e., the "medium") were isolated by dissolving in petroleum naphtha, allowing to stand, chilling and then filtering. Acetone was then added to the extract, alllowed to stand filtered and the extract evaporated to dryness.

Of all the substances tested, Trinidad asphalt showed the highest d(S.T.)/d Temp. value. This undoubtedly accounts for its greater miscibility with coal-tar products, than proves to be the case with petroleum asphalts—even the softer varieties.

The oily constituents show a lower surface-tension than the original substance, at any given temperature. The greatest difference is noted in the case of Trinidad asphalt; coal-tar pitch comes next; and petroleum asphalts come last.

Nellensteyn's bubble method has been found most satisfactory for testing tars between 80 and 90° C., whereas the Du Noüy tensiometer with a platinum ring of 4-cm. periphery gives the most

satisfactory results at room temperatures. Nellensteyn's observations when plotted at different temperatures show a sudden break in the surface-tension readings at lower temperatures, due no doubt to the interfering effect of high viscosities. The method may be simplified as follows:[88]

The tar is brought into sample tube *S* and immersed in a constant-temperature bath (Fig. 234). After the desired temperature equilibrium has been reached, the glass tube, *G*, provided with a fine opening at the end, is gently lowered until its tip touches the surface

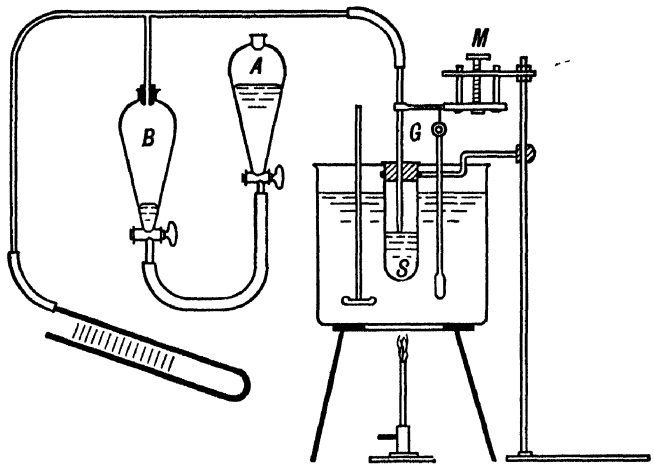


FIG. 234.—Modified Apparatus for Surface-Tension.

of the tar. Contact is readily obtained by use of the screw arrangement, *M*. Then, the two stopcocks are adjusted so that the mercury in container *A* flows slowly into container *B*. Since the air in *B* is gradually displaced by mercury, pressure is developed in the system. At a definite maximum pressure the air bubble formed on the tip of the glass tube bursts, thereby releasing part of the pressure in the system. This maximum pressure is a direct measure of the surface tension of the liquid under consideration.

Since an inclined gage is used, an over-all constant for the apparatus should be ascertained by measuring the maximum bubble pressure for water, nitrobenzene and benzene. In the case of tars, it is advisable to reduce the air displacement to not exceeding two bubbles per minute.

Test 12e. Interfacial Tension. Interfacial tension between bituminous materials and aqueous media is an important consideration in studying dispersions. Various methods have been suggested, but the most accurate has been devised by P. L. Du Noüy,[89] known as the "tensiometer." The procedure is as follows:

The bath used to maintain the temperature for the measurements with the tensiometer, consists of a large crystallizing dish held in place on a plate 18 cm. in diameter by upright arms of spring steel. The plate is fastened to the table of the tensiometer by means of bolts. The dish is filled to about three-fourths capacity with Nujol and equipped with an electrical heating element, thermometer, and mechanical stirrer. An 8-cm. Pyrex crystallizing dish, in which were placed the water or aqueous solution and the asphalt to be investigated, is held in a rigid clamp which fitted to the side and has legs resting on the bottom of the bath. The temperature of the bath is maintained at approximately 85° C. A change of 2° or 3° C. in the temperature has no measurable effect on the values of the interfacial tension.

Since the asphaltic materials have a density slightly lower than that of the water or solution, the latter is placed in the small crystallizing dish, and the platform of the tensiometer raised until the ring is immersed. The asphalt heated to about 85° C. is then carefully poured on the surface of the aqueous phase. After the materials have reached the desired temperature, the ring is pulled slowly upward against the interface, and the platform lowered to keep the pointer on the index line. Three to four minutes are consumed in changing the reading on the dial by one dyne. When the ring breaks through the interface, the reading on the dial is recorded. The instrument is calibrated frequently to indicate the interfacial tension directly in dynes per centimeter.

Two different types of asphaltic materials were investigated. They are distinguished by the letters *A* and *B*. Flux *A* was derived from a Venezuelan asphaltic petroleum by steam distillation, and flux *B* from a Trinidad asphaltic petroleum by the same process; their physical properties being as follows:

	Flux A	Flux B
Sp. gr. at 85/85° C.	0.9964	0.9970
Float at 150° F. (65.6° C.), seconds	44	53
Flash (Cleveland open cup), ° F. (° C.)	420 (215.6)	415 (212.8)

The values obtained using fluxes *A* and *B* and sodium hydroxide solutions from 0.0013 to 0.25 *M* are as follows:

NaOH Concn., Mole/liter	Interfacial Tension Flux A, Dynes/cm.	Flux B Dynes/cm.	NaOH Concn., Mole/liter	Interfacial Tension Flux A, Dynes/cm.	Flux B, Dynes/cm.
0.0000	16.7	15.7	0.0100	0.5	0.0*
0.0013	11.4	7.9	0.0500	0.0*	0.0*
0.0025	9.5	5.3	0.1000	0.0*	0.0*
0.0050	5.3	0.0	0.2500	0.0*	0.0*

* Emulsification apparently took place at the interface. The aqueous solution contained dispersed material which in time made the solution opaque.

These data indicate that flux *B* contained material more reactive toward sodium hydroxide than flux *A*, the resulting compounds causing the lowering of the interfacial tension.

(C) THERMAL TESTS

THERMAL CONDUCTIVITY

The following symbols and equations have been proposed [90] for designating heat transmission:

Area ..... A

Temperature, deg. Fahr. or deg. Cent ..... t

Temperature, deg. Fahr. absolute or deg. K (deg. Cent. absolute) ..... T

Length of path of heat flow (thickness) L

Total quantity of heat transferred ..... Q

Time (when t is used for temperature) ..... τ

Thermal transmission (heat transferred per unit time) ..... q

$$q = \frac{Q}{\tau}$$

Thermal conductivity (heat transferred per unit time per unit area, and per degree per unit length) ..... k

$$k = \frac{\frac{q}{A}}{\frac{(t_1 - t_2)}{L}}$$

Thermal resistivity ..... $\frac{1}{k}$

Thermal resistance (degrees, per unit of heat transferred per unit time) ..... R

$$R = \frac{t_1 - t_2}{q} = \frac{L}{kA}$$

Thermal conductance (heat transferred per unit time, per degree)..... C

$$C = \frac{1}{R} = \frac{kA}{L} = \frac{q}{t_1 - t_2}$$

Thermal conductance per unit area, sometimes called "Unit Conductance" (heat transferred per unit time per unit area, per degree)..... C_A

$$C_A = \frac{C}{A} = \frac{1}{RA} = \frac{q}{A(t_1 - t_2)} = \frac{k}{L}$$

Surface coefficient of heat transfer, film coefficient of heat transfer, individual coefficient of heat transfer (heat transferred per unit time, per unit area, per degree)..... h

$$h = \frac{\frac{q}{A}}{t_1 - t_2}$$

(In general h is not equal to $\frac{k}{L}$, where L is the actual thickness of the fluid film.)

Over-all coefficient of heat transfer, thermal transmittance per unit area (heat transferred per unit time per unit area, per degree over-all)..... U

$$U = \frac{\frac{q}{A}}{t_1 - t_2}$$

The thermal conductivity units in Table XCIV are written in the form:

$$\frac{\text{Heat energy transfer per unit area in unit time}}{\text{Temperature difference per unit length}}$$

A thermal conductivity expressed in any of the units designated in the left-hand column can be converted into any of the units designated in the headings of the columns by *multiplying* ($\times$) by the number which is common to the row and column.

A thermal conductivity expressed in any of the units designated in the headings of the columns can be converted into any of the

units designated in the left-hand column by *dividing* (÷) by the number which is common to the row and column.

Examples:

$$10 \frac{\text{B.t.u. per sq. ft. per sec.}}{\text{deg. Fahr. per in.}} = 10 \times 5.190 = 51.90 \frac{\text{watt per sq. cm.}}{\text{deg. Cent. per cm.}}$$

or

$$10 \frac{\text{B.t.u. per sq. ft. per sec.}}{\text{deg. Fahr. per in.}} = \frac{10}{0.1927} = 51.90 \frac{\text{watt per sq. cm.}}{\text{deg. Cent. per cm.}}$$

TABLE XCIV

CONVERSION FACTORS FOR VARIOUS THERMAL CONDUCTIVITY UNITS

	Cal. per sq. cm. per sec. / deg. Cent. per cm. ÷	Kilocalorie per sq. m. per hr. / deg. Cent. per m. ÷	Watt per sq. cm. / deg. Cent. per cm. ÷	B.t.u. per sq. ft. per sec. / deg. Fahr. per in. ÷	B.t.u. per sq. ft. per hr. / deg. Fahr. per in. ÷	B.t.u. per sq. ft. per hr. / deg. Fahr. per ft. ÷
Cal. per sq. cm. per sec. / deg. Cent. per cm. ×	1	360.0	4.186	0.8064	2903	241.9
Kilocalorie per sq. m. per hr. / deg. Cent. per m. ×	0.002778	1	0.01163	0.002240	8.065	0.6720
Watt per sq. cm. / deg. Cent. per cm. ×	0.2389	86.00	1	0.1926	693.5	57.79
B.t.u. per sq. ft. per sec. / deg. Fahr. per in. ×	1.240	446.4	5.191	1	3600	300
B.t.u. per sq. ft. per hr. / deg. Fahr. per in. ×	0.0003445	0.1240	0.001442	0.0002778	1	0.08333
B.t.u. per sq. ft. per hr. / deg. Fahr. per ft. ×	0.004134	1.488	0.01730	0.003333	12.00	1

NOTE.—Mean calories, mean British thermal units, and absolute watts are used.

The thermal conductivity of bituminous substances may be calculated from the following formula:

$$K = \frac{0.813}{d} [1 - 0.0003(t - 32)]$$

where K = thermal conductivity in B.T.U. per hr., sq. ft., and °F. per in.; d = specific gravity at 60°/60° F.; and t = temperature in °F.

The following figures have been reported for "K";[91]

Petroleum asphalts.....1.2 for temperature range, 32° F. to fusing point.
Filled paving asphalts.....4.5 to 5.2 for temperature range 50 to 90° F.
Paraffin wax.....1.6 for temperature range 32° F. to melting-point.

Example:—The opposite faces of a slab of petroleum asphalt, 3 inches thick, are maintained at 32° and 77° F., respectively. What is the heat flow per day through each square foot of the slab?

The result is obtained as follows: $1.2 \times 24 \times 1 \times \frac{45}{3} = 432$ B.t.u.

Test 12f. A.S.T.M. Method. The following procedure has been standardized[92] for comparing the thermal conductivity of solid materials which are in the form of flat sheets; an accuracy of 10 per cent being obtained:

The thermal conductivity of a homogeneous material is the rate of heat flow under steady conditions, per unit area, and per unit temperature gradient in the direction perpendicular to the area. Thermal conductivity is expressed in watts per square centimeter per degree centigrade per centimeter.

The principle of the method is analogous to the potentiometer method of comparing electrical resistances. A specimen of unknown conductivity is placed in series with a standard specimen of known conductivity between plates which are maintained at different temperatures. When a steady state of heat flow is attained, the respective temperature differences across the standard and the test specimens are measured and the conductivity of the test specimen is calculated from the following relation.

$$K = K_1 \frac{L}{L_1} \times \frac{\Delta t_1}{\Delta t}$$

where K = the conductivity of the test specimen;
K_1 = the conductivity of the standard specimen;
L = the thickness of the test specimen;
L_1 = the thickness of the standard specimen;
Δt = the temperature difference of the test specimen;
Δt_1 = the temperature difference of the standard specimen.

The apparatus, consisting of a heating plate, a cooling plate, three or four thermocouples, a calibrated galvanometer or a thermocouple potentiometer, an ice bath, and at least one standard specimen, shall conform to the following requirements:

(*a*) *Plates.* The heating and the cooling plates shall be flat pieces of metal. The contact surface of each plate shall have dimensions equal to or greater than the contact surface of the specimen. The metal surface which supplies or removes the heat from the specimens shall be maintained at a temperature which is constant and uniform within 0.1° C.

NOTE.—The heating plate may be heated either electrically or by the vapor from a liquid boiling at a definite temperature. The cooling plate may be cooled by a liquid circulating through it. A platen from a steam-heated press makes a satisfactory heating or cooling plate. A plate of this description is shown in Fig. 235.

(*b*) *Thermocouples.* Thermocouples shall be made from wire not larger than 0.25 mm. (0.010 in.) in diameter, or No. 30, A. w. g., one wire being of copper and the other of a suitable alloy which will give an electromotive force of about 40 microvolts per degree Centigrade. The wire shall be provided with suitable insulation of such thickness that the over-all diameter does not exceed 0.50 mm. (0.020 in.). The junctions shall be made by soldering or welding the wires. The measuring junctions of the thermocouples, used for determining the temperatures at the surface of the specimens shall be soldered to or embedded at, the midpoint of sheets of pure tin, or other malleable metal, which have the same shape as the contact surface of the specimens. Each sheet of tin shall not exceed 0.50 mm. (0.020 in.) in thickness, and shall be provided with a slot in which the wires leading to the junction may be laid. No part of the thermocouple or its mounting shall exceed 0.5 mm. (0.020 in.) in thickness. The reference or cold junction of each thermocouple shall be mounted in a glass tube not more than 2.5 mm. (0.1 in.) in diameter, which is sealed at its lower end and is immersed in a bath of crushed ice so that the junction is at least 100 mm. (4 in.) below the surface, and at least 25 mm. (1 in.) above the lower ice level.

NOTE.—The use of a separate reference or cold junction for each thermocouple is essential only when measurements are made on specimens of high graphite content or other materials which may be electrically conducting.

(*c*) *Galvanometer.* If a galvanometer is used on the terminals of the thermocouples it shall have a sensitivity sufficient to indicate definitely a change of temperature of 0.1° C. at the hot junction of any of the thermocouples. The deflections of the galvanometer shall be sufficiently steady and the instrument shall be calibrated to warrant an accuracy of ± 0.25° C.

(*d*) *Potentiometer.* If a potentiometer is used on the terminals of the thermocouples it shall be sufficiently sensitive and precise to warrant an accuracy of ± 0.25° C.

(*e*) *Ice Bath.* The ice bath shall consist of a Dewar flask or other well-insulated vessel filled to a depth of at least 15 cm. (6 in.) with ice and water.

Test specimens shall be in the form of flat sheets which may be square, circular, or rectangular in shape. The specimens shall not project beyond the edges of the heating or cooling plates and the total thickness of a test specimen and a standard specimen shall not exceed one-sixth of the edge of the square, the diameter of the circle or the shortest side of the rectangle. A convenient size for test specimens is approximately 20 cm. (8 in.) square and 1 cm. (0.4 in.) in thickness. Specimens should be uniform in thickness to within ± 1.5 per cent. Thickness measurements shall be made with an accuracy of 0.5 per cent at not less than ten points uniformly distributed over the surface. The average thickness shall be used for computing the thermal conductivity. When the material is soft or easily compressible, the measurement shall be made in such a way as to indicate the thickness under the pressure used in the test.

Standard specimens shall have the same shape and size as the test specimens. Any test in which the conductivity of the test specimen is found to be less than one-half or more than twice the conductivity of the standard specimen to which it was compared shall be tested again using another standard specimen having a conductivity nearer that of the specimen under test. Standard specimens may be made from materials the thermal conductivity of which is reasonably permanent and unaffected by changes in atmospheric humidity. Standard specimens shall be certified, by a standardizing laboratory, for thermal conductivity with an accuracy of ± 2 per cent.

The heating and the cooling plates and the specimens shall be placed in a horizontal position, one on top of the other. The order of assembly from the top down shall be as follows:

(1) Heating plate;
(2) Soft rubber sheet;
(3) Measuring junction of a thermocouple;
(4) Test specimen;
(5) Measuring junction of a thermocouple;
(6) Thin, soft rubber sheet;

(7) Measuring junction of a thermocouple;
(8) Standard specimen;
(9) Measuring junction of a thermocouple;
(10) Soft rubber sheet;
(11) Cooling plate.

The pressure on the specimens shall be such that doubling the pressure does not change the apparent thermal conductivity by as much as 2 per cent. The assembled plates and test specimens shall be enclosed on all sides by at least 2 in. of good thermal insulating material in order to minimize the heat loss at the edges.

NOTE.—The soft rubber sheets called for in the above assembly are for the purpose of insuring good thermal contact at hard surfaces. Soft sheet rubber known as "dental dam" is a suitable and readily obtainable sheet for this purpose. One or more layers may be used at each surface to give the desired cushioning effect. When the specimens are soft and flexible these rubber sheets may be omitted, as may also one of the measuring junctions. Pressures of the order of 200 g. per sq. cm. (400 lb. per sq. ft.) are usually sufficient to insure good thermal contact of soft rubber specimens.

Temperature of the Plates. The heating and the cooling plates shall be maintained at temperatures which are constant within ± 0.1° C. until the temperature differences across the specimens being compared are constant within ± 0.25° C. for one hour, or ± 0.10° C. for 30 minutes.

Temperature of Specimens. The mean of the temperatures at the two surfaces of the test specimen shall be considered the temperature of the specimen and shall be within ± 10° C. of the temperature at which it is desired to know the thermal conductivity. Similarly, the mean of the temperatures at the two surfaces of the standard specimen shall be considered its temperature.

The temperature of the standard specimen shall be within ± 10° C. of a temperature at which its thermal conductivity is known. The temperature difference across any specimen shall not be greater than 50° C. nor less than 10° C.

The thermal conductivity of the test specimen shall be calcuated by the formula given above.

NOTE 1.—The thermal conductivity of most materials increases with increasing temperature. The temperature coefficient near room temperature is usually within the range of 0.002 and 0.005 per 1° C. The change of thermal conductivity with temperature is usually near enough to a linear relation to permit the mean temperature of a specimen to be used even when large temperature differences exist.

NOTE 2.—Temperatures in the specimens being compared may be made approximately those desired by the selection of appropriate temperatures for the heating and the cooling

plates, and by the insertion of sheets of thermal insulating material in the test assembly. The total thickness of these accessory sheets and of the specimens being compared should not exceed the thickness specified as the total thickness of a test specimen and a standard specimen.

NOTE 3.—A temperature of 70° C. is recommended as the temperature at which the thermal conductivity of electrical insulating materials should be measured since many such materials are designed for use at 40° C. above room temperature.

The report shall include the following:

(*a*) The standard specimen used;
(*b*) The thickness of the standard specimen;
(*c*) The thickness of the test specimen;
(*d*) The temperatures at the top and bottom of the test specimen;
(*e*) The temperatures at the top and bottom of the standard specimen;
(*f*) The computed conductivity of the test specimen and its mean temperature.

SPECIFIC HEAT

Test 12g. Conventional Method.[91] This may be calculated from the formula:[93]

$$= \frac{1}{\sqrt{d}}(0.388 + 0.00045t)$$

in which c = specific heat in Btu. per pound per ° F., or calories per gram per ° C., d = specific gravity at 60°/60° F., and t = temperature in ° F.

In cases where the asphalt is mixed with various amounts of solids, such as sand, crushed rock, etc., the specific heat of the mixture may be calculated from the following expression:

$$C_m = 0.01[(100 - x)C_a + xC_s]$$

where x = per cent by weight of solids; C = specific heat; and the subscripts "a," "s," and "m" refer to asphalt, solid, and mixture, respectively. The relation: $C_s = 0.18 + 0.00006t$° F., may be used for the solid constituents. Values for C_a are given in Table XCV.

Example:—What is the specific heat at 60° F. of bituminous material containing by weight 15 per cent asphalt and 85 per cent solid materials? Table XCV and the above formulas give:

$$C_m = 0.01\ (15 \times 0.415) + (85 \times 0.184) = 0.22$$

HEAT CONTENT

Test 12h. Conventional Method. This test [91] is run as follows:

The data, given in Table XCVI, on the heat content of asphalts containing various percentages of mineral matter were calculated from the following equation:

$$H_a = (0.388t + 0.000225t^2 - 12.65)\ (1 - 0.01x) + (0.18t + 0.00003t^2 - 5.76)\ 0.01x$$

in which t = temperature, in ° F. and x = per cent, by weight, of mineral matter.

The data given in the column marked "0 per cent" are applicable to the bitumen content of natural asphalts and to petroleum asphalts which usually contain only small amounts of mineral matter. The data given in the columns marked "10 per cent" to "80 per cent," inclusive, are applicable to natural asphalts and to mixtures of natural or petroleum asphalts with known amounts of mineral matter. In using these data, it should be noted that the content of mineral matter, as used here, includes the so-called "free-carbon" content of the asphalt.

Example.—How much heat is required to raise the temperature of asphalt containing 10 per cent of mineral matter from 60° to 400° F.? The result may be obtained from Table XCVI as follows:

Heat content of asphalt at 400° F. = 168 Btu./lb.
Heat content of asphalt at 60° F. = 11 Btu./lb.
Heat required = difference = 157 Btu./lb.

THERMAL EXPANSION

The thermal expansion of asphalts and other forms of bituminous matter may be calculated [91] from the values given in Table XCVII, where V_{60}/V represent volumes at 60° F. occupied by a unit volume at indicated temperatures, t° F. For example, 1 gallon of petroleum asphalt measured at 350° F., will have a volume of 0.9031 gallon at 60° F.

The data given in Table XCVII were calculated from the equation,

$$V_t = V_{60}\ [1 + A\ (t - 60) + B(t - 60)^2]$$

using $A = 0.000341$ and $B = 0.0000001$, which is equivalent to the following:

Temperature Range in ° F.	Mean Coefficient of Expansion
60 to 150	0.00035
60 to 250	0.00036
60 to 350	0.00037
60 to 450	0.00038

These coefficients and the expansions, $(V_{60}/V_t) - 1$, obtained from Table XCVII apply to petroleum asphalts and fluxes in general with an estimated accuracy of 5 per cent, which is equivalent to the following percentage accuracy in the relative volumes, V_{60}/V_t, for various temperature ranges: 0.1 per cent, 0° to 100°; 0.2 per cent, 100° to 200°; 0.4 per cent, 200° to 300°; 0.6 per cent, 300° to 400°; and 0.8 per cent, 400° to 500° F.

Products containing wax, gas bubbles, or nonbituminous materials have expansions which differ from those given, in proportion to the amount present.

Example.—If the volume of a given quantity of petroleum asphalt is 10,000 gallons at 350° F., what is its volume at 60° F.? Calculate from the value given in Table XCVII as follows:

$$V_{60} = 10{,}000 \times 0.9031 = 9031 \text{ gallons at } 60° \text{ F.}$$

TABLE XCV

SPECIFIC HEAT OF ASPHALTS

Temp. °F.	Btu./lb. °F. or cal./g °C.	Btu./gal. °F.	Temp. °F.	Btu./lb. °F. or cal./g °C.	Btu./gal. °F.
0	0.388	3.23	**400**	0.568	4.74
20	.397	3.31	420	.577	4.81
40	.406	3.38	440	.586	4.89
60	.415	3.46	460	.595	4.96
80	.424	3.53	480	.604	5.04
100	.433	3.61	**500**	.613	5.11
120	.442	3.69	520	.622	5.19
140	.451	3.76	540	.631	5.26
160	.460	3.84	560	.640	5.34
180	.469	3.91	580	.649	5.41
200	.478	3.99	**600**	.658	5.49
220	.487	4.06	620	.667	5.56
240	.496	4.14	640	.676	5.64
260	.505	4.21	660	.685	5.71
280	.514	4.29	680	.694	5.79
300	.523	4.36	**700**	.703	5.86
320	.532	4.44	720	.712	5.94
340	.541	4.51	740	.721	6.01
360	.550	4.59	760	.730	6.09
380	.559	4.66	780	.739	6.16
			800	.748	6.24

TABLE XCVI

HEAT CONTENT OF ASPHALT IN BTU./LB.

Temp. °F.	Heat content of asphalts containing 0 to 80 per cent mineral matter, by weight								Temp. °F.
	0	10	20	30	40	50	60	80	
0	−13	−12	−11	−11	−10	− 9	− 8	−7	0
10	− 9	− 8	− 8	− 7	− 7	− 6	− 6	− 5	10
20	− 5	− 5	− 4	− 4	− 4	− 3	− 3	− 3	20
32	0	0	0	0	0	0	0	0	32
40	+ 3	+ 3	+ 3	+ 3	+ 2	+ 2	+ 2	+ 2	40
50	7	7	6	6	6	5	5	4	50
60	11	11	10	10	9	8	8	6	60
70	16	15	14	13	12	11	10	9	70
80	20	19	18	17	15	14	13	11	80
90	24	23	21	20	19	17	16	13	90
100	28	27	25	24	22	21	19	16	100
110	33	31	29	27	25	24	22	18	110
120	37	35	33	31	29	27	25	20	120
130	42	39	37	35	32	30	28	23	130
140	46	43	41	38	36	33	30	25	140
150	51	48	45	42	39	36	33	28	150
160	55	52	49	46	43	40	36	30	160
170	60	57	53	50	46	43	39	33	170
180	65	61	57	53	50	46	42	35	180
190	69	65	61	57	53	49	45	37	190
200	74	70	66	61	57	53	48	40	200
210	79	74	70	65	61	56	52	42	210
220	84	79	74	69	64	60	55	45	220
230	89	83	78	73	68	63	58	47	230
240	93	88	83	77	72	66	61	50	240
250	98	93	87	81	75	70	64	52	250
260	103	97	91	85	79	73	67	55	260
270	108	102	96	89	83	77	70	58	270
280	114	107	100	94	87	80	74	60	280
290	119	112	105	98	91	84	77	63	290
300	124	117	109	102	95	87	80	65	300
310	129	122	114	106	99	91	83	68	310
320	135	127	119	111	103	95	87	71	320
330	140	132	123	115	107	98	90	73	330
340	145	137	128	120	111	102	94	76	340
350	151	142	133	124	115	106	97	79	350
360	156	147	138	128	119	110	100	82	360
370	162	152	142	133	123	113	104	84	370
380	167	157	147	137	127	117	107	87	380
390	173	163	152	142	131	121	111	90	390
400	179	168	157	146	136	125	114	93	400
420	190	178	167	155	144	133	121	98	420
440	202	189	177	165	153	140	128	104	440
460	213	200	187	174	161	148	135	109	460
480	225	212	198	184	170	156	143	115	480
500	238	223	208	194	179	165	150	121	500

TABLE XCVII

Thermal Expansion of Petroleum Asphalts and Fluxes

Temp. t°F.	$\frac{V_{60}}{V_t}$	Temp. t°F.	$\frac{V_{60}}{V_t}$	Temp. t°F.	$\frac{V_{60}}{V_t}$	Temp. t°F.	$\frac{V_{60}}{V_t}$	Temp. t°F.	$\frac{V_{60}}{V_t}$
0	1.0205	100	0.9864	200	0.9527	300	0.9195	400	0.8869
2	1.0198	102	.9857	202	.9520	302	.9188	402	.8863
4	1.0191	104	.9850	204	.9513	304	.9181	404	.8856
6	1.0185	106	.9844	206	.9506	306	.9175	406	.8850
8	1.0178	108	.9837	208	.9500	308	.9168	408	.8843
10	1.0171	110	.9830	210	.9493	310	.9162	410	.8837
12	1.0164	112	.9823	212	.9486	312	.9155	412	.8831
14	1.0157	114	.9816	214	.9480	314	.9149	414	.8824
16	1.0150	116	.9810	216	.9473	316	.9142	416	.8818
18	1.0143	118	.9803	218	.9466	318	.9135	418	.8811
20	1.0137	120	.9796	220	.9460	320	.9129	420	.8805
22	1.0130	122	.9789	222	.9453	322	.9122	422	.8799
24	1.0123	124	.9783	224	.9446	324	.9116	424	.8792
26	1.0116	126	.9776	226	.9440	326	.9109	426	.8786
28	1.0109	128	.9769	228	.9433	328	.9103	428	.8779
30	1.0102	130	.9762	230	.9426	330	.9096	430	.8773
32	1.0095	132	.9755	232	.9420	332	.9090	432	.8767
34	1.0089	134	.9749	234	.9413	334	.9083	434	.8760
36	1.0082	136	.9742	236	.9406	336	.9077	436	.8754
38	1.0075	138	.9735	238	.9400	338	.9070	438	.8747
40	1.0068	140	.9728	240	.9393	340	.9064	440	.8741
42	1.0061	142	.9722	242	.9386	342	.9057	442	.8735
44	1.0054	144	.9715	244	.9380	344	.9051	444	.8728
46	1.0048	146	.9708	246	.9373	346	.9044	446	.8722
48	1.0041	148	.9701	248	.9367	348	.9038	448	.8716
50	1.0034	150	.9695	250	.9360	350	.9031	450	.8709
52	1.0027	152	.9688	252	.9353	352	.9025	452	.8703
54	1.0020	154	.9681	254	.9347	354	.9018	454	.8697
56	1.0014	156	.9674	256	.9340	356	.9012	456	.8690
58	1.0007	158	.9668	258	.9333	358	.9005	458	.8684
60	1.0000	160	.9661	260	.9327	360	.8999	460	.8678
62	.9993	162	.9654	262	.9320	362	.8992	462	.8671
64	.9986	164	.9647	264	.9313	364	.8986	464	.8665
66	.9980	166	.9641	266	.9307	366	.8979	466	.8659
68	.9973	168	.9634	268	.9300	368	.8973	468	.8652
70	.9966	170	.9627	270	.9294	370	.8966	470	.8646
72	.9959	172	.9620	272	.9287	372	.8960	472	.8640
74	.9952	174	.9614	274	.9280	374	.8953	474	.8633
76	.9945	176	.9607	276	.9274	376	.8947	476	.8627
78	.9939	178	.9600	278	.9267	378	.8940	478	.8621
80	.9932	180	.9594	280	.9260	380	.8934	480	.8614
82	.9925	182	.9587	282	.9254	382	.8927	482	.8608
84	.9918	184	.9580	284	.9247	384	.8921	484	.8602
86	.9912	186	.9573	286	.9241	386	.8914	486	.8595
88	.9905	188	.9567	288	.9234	388	.8908	488	.8589
90	.9898	190	.9560	290	.9228	390	.8901	490	.8583
92	.9891	192	.9553	292	.9221	392	.8895	492	.8577
94	.9884	194	.9547	294	.9214	394	.8888	494	.8570
96	.9878	196	.9540	296	.9208	396	.8882	496	.8564
98	.9871	198	.9533	298	.9201	398	.8876	498	.8558
100	.9864	200	.9527	300	.9195	400	.8869	500	.8552

The following coefficients of expansion per ° C. have been reported for various asphaltic products: [94]

	Temperature Range	Coefficient of Expansion
Asphalt saturant for prepared roofing (fusing-point 34° C. K. and S.)	15–60° C.	0.00061
Asphalt coating for prepared roofing (fusing-point 73° C. K. and S.)	15–60° C.	0.00059
Residual petroleum asphalt (fusing-point 81° C.)	15–60° C.	0.00062
Refined Trinidad asphalt	15–60° C.	0.00046

Additional figures applicable to asphalts, tars and pitches are given in the following table: [95]

TABLE XCVIII

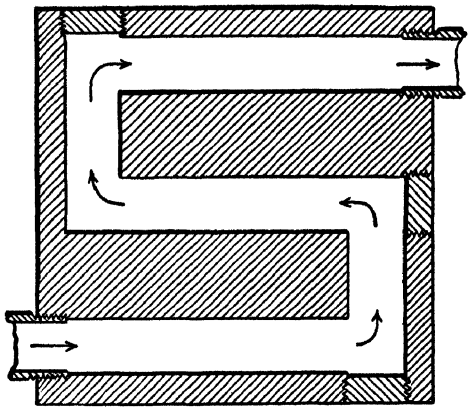
Material		Temperature Range, deg. Fahr.	Coefficient of Expansion per 1 F.	
			Coal Tar	Water-gas Tar
Crude tars	Horizontal coal tar	60 to 150	0.00027 to 0.000305	
	Vertical coal tar	60 to 150	0.000355 to 0.00037	
	Coke oven tar	60 to 150	0.00032 to 0.00034	
	Low temperature tar	60 to 150	0.00042	
	Light water-gas tar	60 to 150	0.00035 to 0.00037	
	Heavy water-gas tar	60 to 150	0.00031 to 0.00032	
Tar road materials and pitch	Cutback products	60 to 180	0.00033 to 0.00037	0.00034 to 0.00035
	Patching materials	60 to 180	0.00033	0.00328 to 0.00034
	Hot application	100 to 200	0.0003 to 0.00035	0.00029 to 0.00032
	Cements	100 to 300	0.0003	0.0003
	Pitches	200 to 300	0.000255 to 0.00028	0.000255 to 0.00028
Asphaltic road material	Road oils	60 to 180	0.0004 to 0.00041	
	Cutbacks	60 to 180	0.0004 to 0.00042	
	Hot application	200 to 300	0.00036	
	Cements	200 to 350	0.00033 to 0.00034	
Solid asphalts		32 to 140	0.00035 to 0.00039	
		60 to 250	0.00033 to 0.00039	
		200 to 400	0.00033 to 0.00035	
		250 to 350	0.000362 to 0.000384	
		350 to 450	0.000378 to 0.000399	
		60 to 450	0.00036 to 0.000382	
Petroleum asphalts and fluxes		60 to 150	0.00035	
		60 to 250	0.00036	
		60 to 350	0.00037	
		60 to 450	0.00038	

Test 12i. A.S.T.M. Method. The following method has been standardized [96] for ascertaining the coefficient of expansion of bituminous compounds:

NOTE.—Certain materials when cooled form voids within the mass and no satisfactory method has been found to measure their coefficient of expansion.

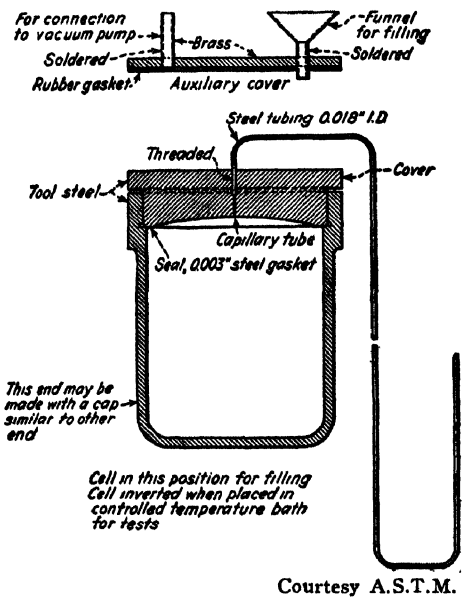
This method is intended for determining the true and effective

coefficient of expansion of compounds. The true coefficient of expansion is for compounds which are free of entrapped gases. The effective coefficient of expansion is the coefficient for material which



Courtesy A.S.T.M.

FIG. 235.—Cross-section of Heating or Cooling Plate.



Courtesy A.S.T.M.

FIG. 235A.—Metallic Cell for Coefficient of Expansion Determinations.

has not been degasified just prior to test. It is important for many purposes to know the effective coefficient of the material as received or after heating to the maximum temperature of application. Consistent results, however, may only be obtained with gas-free compounds.

Compounds having high viscosity may be degasified by heating in a vacuum oven or in a cell fitted with a special cover with vacuum connection similar to the cover shown in Fig. 235A.

In either method the temperature and vacuum should be high enough and the time long enough to insure the driving off of mechanically entrapped gases, but should tend to decompose the compound as little as possible. A gas-free condition is indicated when bubbles of gas no longer appear at the surface of the compound.

The percentage of entrapped gases may be calculated from the following formula (See also Test 7*f*):

$$\text{Volume, in per cent, of entrapped gases} = \frac{\text{sp. gr. of degasified compound} - \text{sp. gr. of untreated compound}}{\text{sp. gr. of degasified compound}} \times 100$$

The following methods are recommended:

Methods A and B. Methods *A* and *B* for true coefficient of expansion are intended for use only where the uniformity of the material under test justifies a high degree of precision. Method *A* is suitable for testing low-viscosity types such as waxes and petrolatums. Method *B* is suitable for testing asphalts and high-viscosity materials, also for opaque materials which may give difficulty in reading the glass scale of Method *A*.

Methods C, D and E. Methods *C*, *D* and *E* are intended for faster testing where high precision may not be justified. These methods may be used for determining either true or effective coefficient of expansion.

Method A. Using Glass Flask

(*a*) *Flask.* The flask * shall hold approximately 250 ml. to the zero mark, and the neck of the flask shall be 1 cm. in internal diameter and graduated for 25 ml. in 0.1-ml. divisions.

(*b*) *Oil Bath.* The cylindrical oil bath for heating the sample shall be approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth with a false bottom 1 in. from the bottom and shall have provision for circulating and heating the oil.

* A Pyrex or quartz glass flask is very satisfactory for this purpose.

(*c*) *Lead Collar.* A lead collar shall be hung on the flask during test to prevent the oil currents of the bath from moving the flask.

The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

The flask shall be maintained under a vacuum of 25 in. of mercury and a temperature 50° C. higher than the softening point (Ring-and-Ball method) while being filled and for approximately 30 min. after filling is complete. The flask shall be filled to within the last milliliter marked on the neck when held at the maximum test temperature and slowly cooled to room temperature (10 to 12 hr.). Before starting the test, the flask shall be examined for the presence of cavities or irregular contraction of the compound. Some compounds after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In such cases it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask shall be placed in the bath for several hours to insure temperature equilibrium.

With the compound satisfactorily placed in the flask at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 50° C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound shall be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction it may be reduced to 30 min.

After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and expansion readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion of the compound} = \frac{V_1 - V}{(T_1 - T)V} + C$$

where V = the original volume occupied by the compound;
V_1 = the volume at higher temperature occupied by the compound;
T = the original temperature;
T_1 = the higher temperature, and
C = a constant = coefficient of expansion of glass container.

The coefficient of expansion shall be calculated for three temperature ranges, as follows:

(1) From the minimum temperature at which the measurement was made to 10° C. below the melting point. This is intended to give the average coefficient for the solid condition.

(2) From 5° C. above the melting point to 100° C. This is intended to give the average coefficient for the liquid condition.

(3) From the minimum temperature at which a measurement was made to 100° C.

Method B. Using Metallic Cell

(*a*) *Metal Cell.* The cell shall be made of steel, consisting of four parts, a cylinder about 2.5 in. in internal diameter, having a rigid bottom, a metallic gasket, a cover to which a steel capillary tube is attached and an auxiliary cover for introducing the compound to be tested while the cell is under a vacuum. The cell shall have an internal volume of approximately 250 ml.

Figure 235A shows the metallic cell for coefficient of expansion determinations. The cell consists of four principal parts: A steel cylinder, a metallic gasket, a steel cover, and a dummy or auxiliary cover for filling. The gasket must be of a metal which does not amalgamate with mercury.

The cylinder is about 2.5 in. (6.35 cm.) in internal diameter, and approximately 3 in. (7.6 cm.) in internal depth. The top of the cylinder is threaded to receive the steel cover and has a machined shoulder to seat a 0.003 in. thick metallic gasket. The cylinder may be of one piece construction or fitted with a cap at the bottom similar to the top end. The steel cover is carefully rounded on the under side to avoid air pockets. It is threaded into the top of the cylinder and seats on the metallic gasket. The center of the cover is threaded to receive a steel capillary tube of 0.018 in. in

internal diameter. The auxiliary cover is brass with a rubber gasket seated by atmospheric pressure. A brass funnel and vacuum connection are soldered to the cover. A plug is provided for sealing the funnel while the cell is maintained under the vacuum specified after filling.

(*b*) *Oil Bath.* The oil bath shall be the same as described in Method *A*, with the exception that provision shall be made for supporting the metal cell.

The cell shall be calibrated to determine its volume at various temperatures as follows:

(*a*) Weigh the assembled cell to determine its tare weight.

(*b*) Fill the cell with mercury until replacing the cover causes some to extrude through the capillary tubing. Record the weight of the cell and mercury and note the temperature.

(*c*) Place cell in the oil bath in an inverted position. The capillary tubing should extend over the side of the oil bath in such a way that the extruded mercury may be caught in a beaker. The oil bath which is several degrees above room temperature causes some mercury to be extruded from the capillary tube. When all expansion has taken place, weigh the mercury collected.

(*d*) Adjust the oil bath for other test temperatures and note the amounts of mercury extruded. The weight of mercury in the cell at any temperature is thus determined and the volume may be calculated.

The cell shall be fitted with the auxiliary cover and held in an upright position in the oil bath while filling. A vacuum of 25 in. of mercury and a temperature 50° C. higher than the softening point (ring-and-ball method) shall be maintained while filling and for at least 30 min. after the cell has been filled to within approximately ¼ in. of the cover. The cell shall then be slowly cooled to room temperature, and any irregularities in the surface of the compound removed. The auxiliary cover shall be replaced with the standard cover and the cell and compound weighed. Sufficient mercury shall be poured into the cell so that some is extruded when the cover is screwed down. The cell shall then be weighed again. The cell shall then be inverted and placed in the oil bath, and the procedure outlined under calibration, Method *B* again followed for 5° C. intervals. Only clean, distilled mercury shall be used. During the test, temperature readings shall be taken at top and bottom

of the bath to detect any variation. Readings of the expansion of the compound should be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction, it may be reduced to 30 min.

After volumetric determinations have been made over the desired temperature range, a curve shall be plotted between volume and temperature readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion of the compound} = \frac{V_1 - V}{(T_1 - T)V}$$

where V = the original volume occupied by the compound;
V_1 = the volume at higher temperature occupied by the compound;
T = the original temperature, and
T_1 = the final temperature.

The coefficient of expansion shall be calculated for the same three ranges as prescribed in Method *A*.

The report shall include the following:

(*a*) The type of cell used.
(*b*) A copy of the volume-temperature curve.
(*c*) The temperature ranges as prescribed in Method *A*.
(*d*) The coefficient of expansion corresponding to each of the three temperature ranges.

Method C. Specific Gravity Method

The specific gravity of untreated or degasified compound at two test temperatures shall be ascertained by any of the suitable procedures described in Test 7. From the temperature and specific gravity the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{\text{SD. GR. AT } T = \text{SP. GR. AT } T_1}{(T_1 - T)\ \text{SP. GR. AT } T_1}$$

where T = the initial temperature, and
T_1 = the higher temperature.

Method D. Pycnometer Contraction

This method is a modification of the specific gravity method (Method C). It may be applied to either untreated or degasified compounds.

(*a*) *Flask.* A 100-ml. volumetric glass * flask shall be used and the zero mark shall be as near as possible to the bulb of the flask.

(*b*) *Oil Bath.* The oil bath may consist of a tall-form glass * beaker of sufficient size so that when the flask is supported about 1 in. from the bottom the oil level will reach at least to the zero mark of the flask.

(*c*) *Metal Collar.* Lead or iron collars placed on the neck of the flask shall be used during heating to prevent oil currents of the bath from moving the flask.

The flask shall be weighed and then filled to within approximately 10 ml. of the zero mark with the compound to be tested, care being taken that at no time shall the temperature of the compound exceed the softening point (ring-and-ball method) by more than 10° C. Care shall also be taken so that none of the compound remains in the stem of the flask. The flask shall be cooled to room temperature, weighed, immersed in the oil bath and placed in an oven previously heated to the maximum temperature at which the test is to be run. After the oil bath reaches the test temperature it shall be allowed to remain at this temperature for 1 hr. The flask shall be removed from the oven, and mercury from a burette shall be quickly poured into it until the level of the compound is within 0.5 ml. of the zero mark. The quantity of mercury used shall be noted in milliliters. The flask shall be replaced in the oven for 20 min., removed again and the mercury titration shall be continued until the level of the compound is exactly at the zero mark. The number of milliliters of mercury used in both titrations shall be recorded.

The oven temperature shall be reset at the next lower test point, temperature equilibrium allowed for as above, and the titration shall be repeated. The same procedure shall be continued at all subsequent test points, the milliliters of mercury added at each point being recorded.

* Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

To find the coefficient of expansion of the material between any two temperatures, the calculations shall be made as follows:

Volume of mercury A at room temperature corrected to temperature $t = A + 0.000182\,A\,(t - 20)$.

Example.—If volume of flask is 100 ml. at 20° C.; volume at $t = 100 + 0.0025\,(t - 20)$ml.

Let $X = V_1 - M_1$ = the volume of compound at T_1,
Let $Y = V_2 - M_2$ = the volume of compound at T_2,

Then the expansion in cu. cm. per gram, $T_1 - T_2 = \frac{X}{W} - \frac{Y}{W}$

the expansion per cu. cm., $T_1 - T_2 = \frac{\frac{X-Y}{W}}{\frac{Y}{W}} = \frac{X-Y}{Y}$

the coefficient of expansion, cu. cm. per cu. cm. per degree Centigrade. $= \frac{X-Y}{Y\,(T_1 - T_2)}$

where W = the weight of sample in grams
T_1 = the higher temperature in degrees Centigrade
T_2 = the lower temperature in degrees Centigrade
M_1 = milliliters of mercury (corrected) added at T_1
M_2 = the sum of milliliters of mercury (corrected) added at T_1 and at T_2
V_1 = the corrected volume of flask at T_1
V_2 = the corrected volume of flask at T_2

To obtain the curve of expansion, plot the points $\frac{W}{X}$, $\frac{W}{Y}$, etc., which are the densities at various temperatures.

Method E. Pycnometer Expansion

This method is another modification of the specific gravity method (Method *C*) and may also be applied to either untreated or degasified materials.

The pycnometer and bath required are the same as described in Method *D*, except that the neck of the flask shall be cut off at the 100-ml. point and ground square. A metal pycnometer may be used provided its coefficient of expansion is known. In this procedure the pycnometer shall be allowed to cool slowly to the lowest test temperature. During the cooling period the flask shall be kept filled by adding more compound and after equilibrium is reached the excess material shall be removed by passing a sharp, flat blade

over the rim. The flask shall then be removed from the bath and quickly weighed. Knowing the tare weight and volume of the flask, the specific gravity may be determined. For successively higher temperatures, it is only necessary to weigh the extruded portion.

From the temperature and weight readings the coefficient of expansion may be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{W - W_1}{W_1(T_1 - T)} - \frac{WC}{W_1}$$

where W = the initial weight of the compound in the flask
W_1 = the weight of the compound in the flask at higher temperature
T = the initial temperature
T_1 = the higher temperature
C = the coefficient of expansion of the flask

Method F. Plummet Displacement

This method is another modification of the specific gravity method (Method *C*).

The apparatus shall consist of the following:

(*a*) *Balance.* An analytical balance equipped with pan straddle.

(*b*) *Plummet.* An aluminium plummet of suitable shape weighing 5 to 10 g.

(*c*) *Beaker.* A 400-ml. glass * beaker wrapped with asbestos.

(*d*) *Thermometer.* A thermometer of suitable range.

(*e*) *Wire.* Two pieces of fine copper wire.

The specific gravity at pouring temperature shall be calculated from the weight of compound displaced by the calibrated aluminium plummet.

Calibration of Plummet. The following weight determinations of the plummet, to the nearest 1 mg., shall be made:

a = the weight in air;
b = the weight suspended in water at 25° C. (77° F.).

* Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

Then: $a - b$ = the weight of water displacement at 25° C. (77° F.).

The value of the plummet displacement (D_{t_p}) in terms of grams of water at 25° C. (77° F.) shall then be corrected to the pouring temperature, t_p, in Centigrade degrees by means of the following formula:

$$D_{t_p} = 0.000076\ (t_p - 25)\ (a - b) + (a - b)$$

NOTE.—Coefficient of cubical expansion of aluminium per deg. Cent. = 0.000076.

Testing of the Sample. The sample shall be carefully melted in the beaker and the temperature raised to approximately 15° C. (27° F.) above the desired test temperature. The beaker shall be placed on the straddle and the plummet suspended in the compound by the fine copper wire. (The weight of the wire should be balanced by placing an equal weight of the wire on the opposite balance pan.)

The scales shall be balanced approximately and at the same time the sample shall be stirred slowly, using the thermometer as a stirring rod. When the sample has cooled to the desired temperature, the weighing shall be completed rapidly.

Calculation of Specific Gravity, t_p/25° C. The specific gravity shall be calculated from following formula:

$$\text{Sp. Gr., } t_p/25^\circ \text{ C.} = \frac{\text{Weight of Plummet in Air} - \text{Weight of Plummet in Compound}}{D_{t_p}}$$

After the specific gravity of the sample at 25° C. (77° F.), and at pouring temperatures have been determined according to the above described methods, the coefficient of expansion shall be calculated according to the formula given in Method *C*.

BREAKING POINT

Various devices have been recommended for ascertaining the breaking-point of asphalts and pitches.[87] They all are based upon determining the temperature at which a layer of predetermined thickness assumes a definite degree of brittleness. The test is of value in determining the temperature range at which bituminous materials in a comparative thin layer will remain pliable to the extent of bending without breaking or fracturing.

Test 13a. Knife Test. The following simple test has been standardized[96] for ascertaining the breaking-point of bituminous compositions: The substance shall be spread on a piece of sheet metal in a layer 1/16 in. in thickness. It shall then be submerged in water having a temperature at least 20° F. above the expected breaking-point. It shall then be cooled 5° F. and held at this temperature for 5 minutes. The cooling shall then be continued in increments of 5° F. At each temperature interval the substance shall be tested with a blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable, and pulling the substance from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the substance no longer stretches but snaps, shall be considered the breaking-point.

Test 13b. Reeve and Yeager's Method. The following procedure has been proposed by Reeve and Yeager:[98] A standard test piece of bituminous material 1/4 in. thick, 1 in. wide, and 4 in. long, is prepared by melting at as low a temperature as possible to avoid volatilization and then pouring into a brass mold coated with mercury. The specimen is carefully removed and inserted into the clamp of the apparatus illustrated in Fig. 310. The water bath is maintained at a temperature somewhat above the estimated breaking-point for fifteen minutes, whereupon the sample is bent around the mandrel of 3/16-in. radius, through an arc of 180 deg. in exactly ten seconds, using a metronome. If not broken, another specimen is inserted in the clamp and the temperature of the bath lowered 10° F. and the test repeated. In this way, working between limits and gradually narrowing them, a point is reached at which a change of 2° F. shows a break or no break. The lower temperature is then reported as the "breaking-point" of the material under examination.

Test 13c. Fraas Method. The following test has been proposed by Ing. Fraas,[99] in which the breaking-point represents the temperature at which a layer of the substance 0.5 mm. thick affixed to a sheet of steel, will crack when bent over a mandrel 9 mm. in diameter in one second time. The prescribed quantity of substance for each test is 0.4 ml., which may either be weighed directly on the steel strip, or extruded from the small press "*A*" shown in Fig.

236, the details of which are illustrated in Fig. 237, of which the mold is cylindrical in form, measuring 20 mm. in diameter by 20

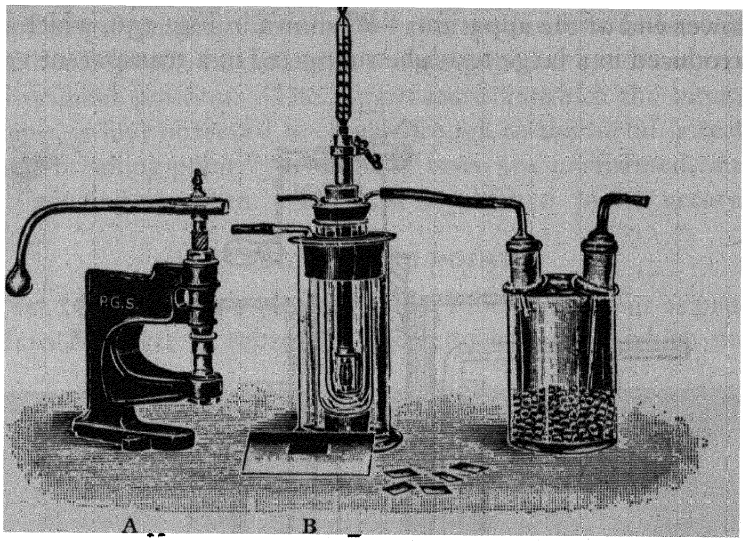


FIG. 236.—Fraas Breaking-point Apparatus.

mm. in height, having a slit in the bottom 20 mm. long by 0.5 mm. wide, through which is extruded a 0.4 ml. strip of standard dimen-

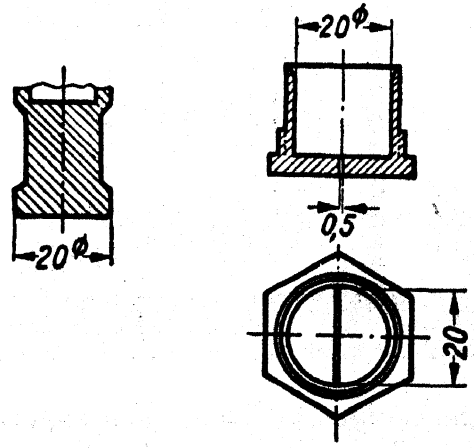


FIG. 237.—Mold of Fraas Breaking-point Apparatus.

sions. The strip is applied to a sheet of tern steel 40 mm. by 20 mm. by 0.15 mm. and heated at 105–110° C. until air and moisture is

expelled and proper adhesion to the metal is secured. Any minute air bubbles are removed from the surface by the rapid passage of a gas flame over same. When cool, the strip is inserted in the clip at the lower end of the apparatus "*B*" shown in Fig. 236, which in turn is introduced in a large test-tube supported in a transparent vacuum-

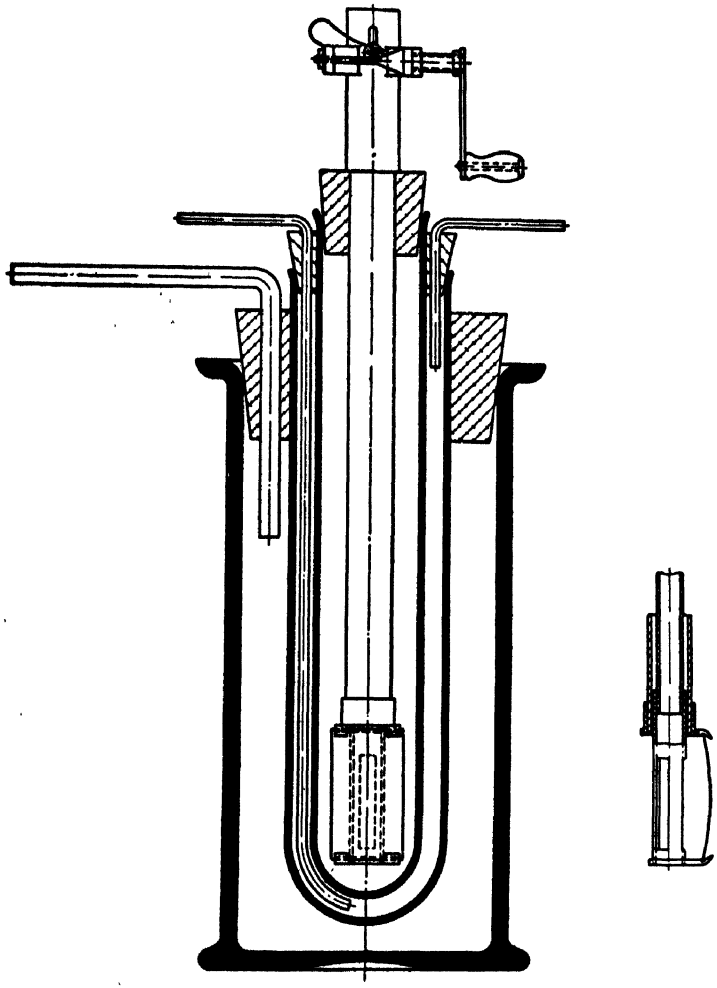


FIG. 238.—Fraas Breaking-point Tester Shown Sectionally.

flask containing the cooling medium. The apparatus as assembled is illustrated sectionally in Fig. 238. Cooling may be effected by drawing dry air through ether, or for lower temperatures (i.e., to minus 20° C.) through a mixture of solid CO_2 with alcohol or ether. The rate of temperature fall is carefully adjusted to 1° C.

per minute, and the specimen is bent at each degree drop in temperature (commencing at 10° C. above the expected breaking-point) by turning the handle at the top of the apparatus, at a speed of one revolution per second. This causes the specimen to be bent around the mandrel 9 mm. in diameter, whereupon the handle is returned to its original position. The temperature at which the bituminous substance is first observed to crack through to the metal is recorded as the "breaking-point." Duplicate tests are performed, and the lower reading is taken. The test is claimed to be accurate to ± 1° C.

SOLIDIFYING-POINT

Test 14. Metzger's Method. The solidifying-point as proposed by Hans Metzger [100] represents the temperature at which the sub-

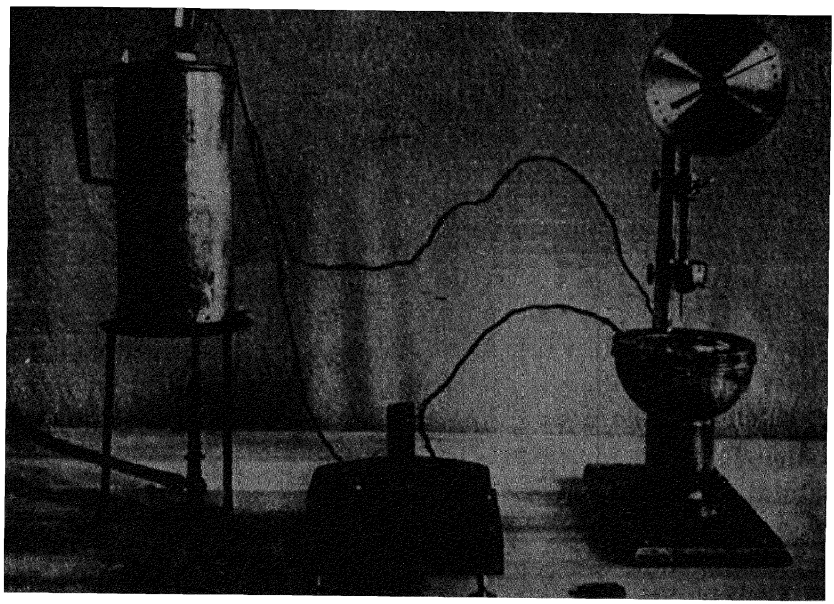


FIG. 239.—Metzger Solidifying-point Tester Assembled.

stance upon cooling attains a definite degree of solidity (i.e., hardness). Specifically, it represents the temperature at which a cylindrical plunger 0.5 mm. in diameter will penetrate the bituminous substance exactly 0.1 mm. under a load of 450 g. in 60 seconds. The apparatus for ascertaining the solidifying-point is illustrated

in Figs. 239, 240 and 241, and is composed of a plunger "q" having a flat bottom 0.5 mm. in diameter, attached to the lower end of a shaft "n," which with the weight "o" aggregates 450 g. The motion of the shaft is measured by the milled rod "c," connected by suitable gears in the housing "b" with the pointer "k" on the movable scale "i," graduated in degrees and tenths of a degree, corresponding to penetrations of 0.1 mm. and 0.01 mm. respectively. The shaft is held in position by the winged screw "m" actuating

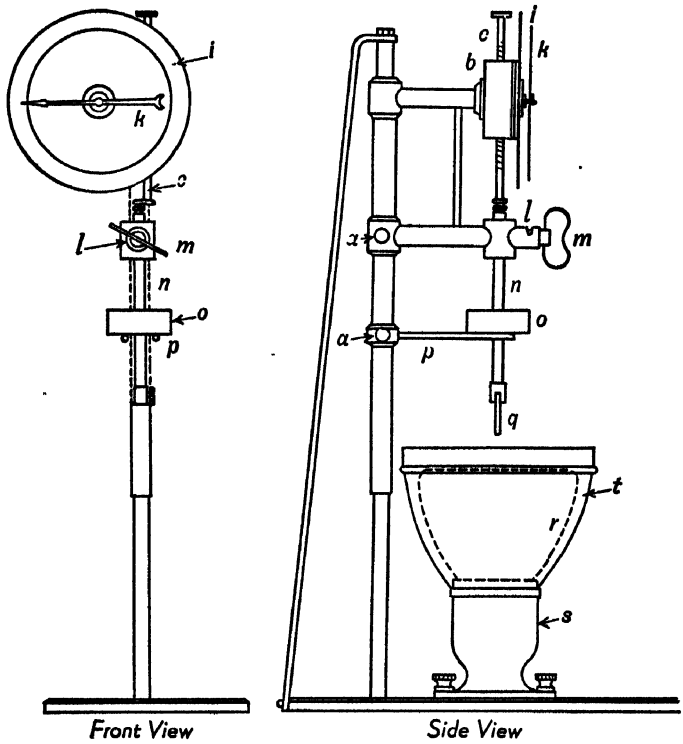


FIG. 240.—Metzger Solidifying-point Tester Shown Sectionally.

against the clamp "l." The fork "p" is used to support the weight and attached shaft when the instrument is not in use.

The substance to be tested is melted at the lowest possible temperature and poured into the receptacle "u," measuring 1.5 cm. wide, 3 cm. long and 2 mm. deep. Before it solidifies, the junction of a thermo-couple composed of iron and Constantine wires is embedded below the surface of the substance, in which it is caused to protrude 1 cm. The receptacle is supported horizontally upon be-

ing slid on the runners of the support "v" cemented inside a Dewar vessel "r," enclosed in a metal jacket "t," which in turn is attached to the base "s." One end of the thermo-couple, together with a thermometer, is immersed in paraffin oil maintained at a constant temperature of 35° C. in a stoppered Thermos flask, which in turn is enclosed in a metal vessel heated with a gas flame, regulated by a thermostat, the purpose of which is to prevent radiation from the Thermos flask. The free ends of the wires are connected with a

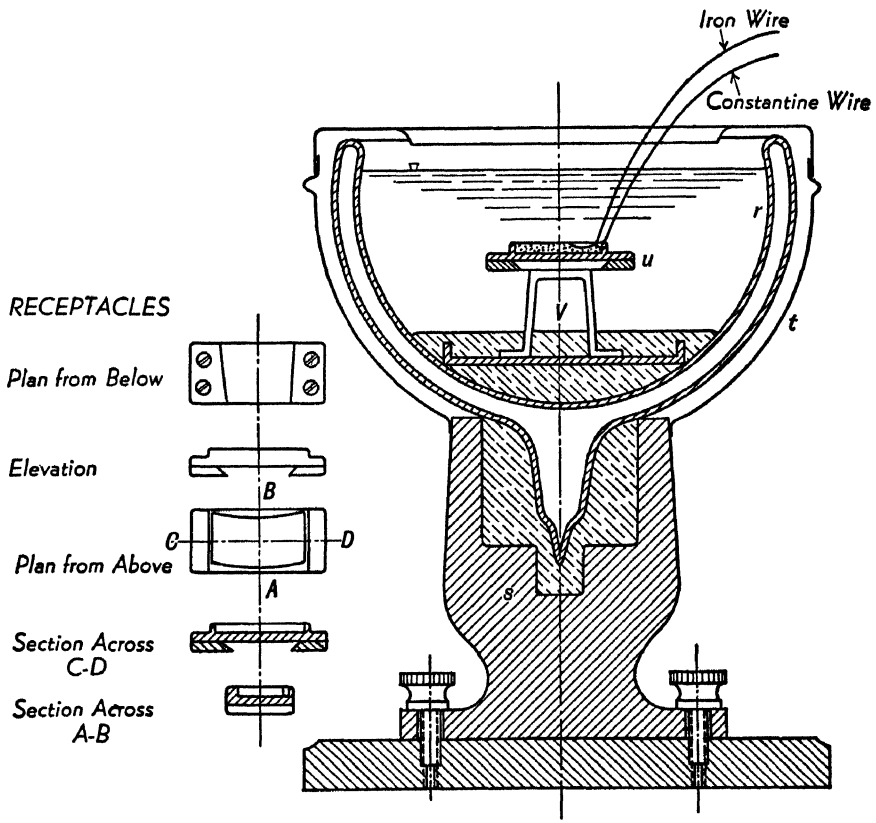


FIG. 241.—Details of Receptacle—Metzger's Solidifying-point Tester.

galvanometer (198 ohms) reading to 5 millivolts, which under the prescribed conditions will record temperatures as low as −70° C. The galvanometer readings should be accurately calibrated against a pentane thermometer in tenths of a degree.

The Dewar vessel "r" is thereupon filled with refrigerated alcohol to within 1 cm. of its upper rim, which is further cooled by

stirring in solidified carbon-dioxide in powdered form until the temperature of the substance is brought about 10° C. lower than the estimated solidifying-point. When the galvanometer reading remains substantially constant, the measuring device is lowered by releasing the screws "a" until the plunger "q" is supported within 2 mm. of the surface of the substance. Then by releasing the winged screw "m," the plunger is carefully lowered until it rests against its surface. The rod "c" is thereupon depressed until its lower foot is brought in contact with the shaft, when the scale "i" is rotated until set at the zero point. Using a stop-watch, the shaft is suddenly released and the plunger allowed to penetrate the substance for exactly 60 seconds, during which interval it is maintained within a temperature range of 1 to 2° C., whereupon a reading is taken to ascertain the depth of penetration.

The temperature of the alcohol is raised slightly, by stirring or blowing air through it, and when it becomes constant, another penetration reading is taken 2 to 3 mm. from the first spot (upon rotating the Dewar vessel). In this manner, a series of temperature and penetration readings are obtained, until the penetration finally measures 0.3 to 0.4 mm. From these readings a graph is constructed on coordinate paper, in which the penetration depths are recorded as abscissas and the temperature as ordinates. By interpolation, the temperature is noted at which the penetration equals exactly 0.1 mm., and this temperature is recorded as the "solidifying-point."

Metzger has worked out some interesting relationships between the solidifying-point representing the temperature at which the bituminous substance attains a *definite* degree of solidity, and Ubbelohde's liquefying-point (Test 15h)—representing the temperature at which the substance attains a definite degree of liquidity (i.e., softness).

These tests are taken as the two extremes, representing fixed points in the consistency range of bituminous substances. It is claimed that the temperature range between the liquefying-point and solidifying-point constitutes a numerical index for each bituminous substance, representing its resistance to temperature changes. The higher this index, the more resistant will the substance be, and conversely, the lower this index, the more rapidly will the substance

pass from the solid into the liquid state upon being subjected to increasing temperatures. The indices applicable to typical substances are given in Table XCIX:

TABLE XCIX

RELATIONSHIP BETWEEN LIQUEFYING AND SOFTENING-POINTS.

	Softening-point		Liquefying-point	Solidifying-point	Temperature Range
	R. and B.	K. and S.			
Montezuma 1.....	66.0° C.	52.0° C.	77.0° C.	− 6.5° C.	83.5° C.
Montezuma 3.....	58.0	43.0	69.0	−17.6	86.6
Mexpetebano 1.....	65.0	49.5	75.0	− 7.9	82.9
Mexpetebano 3.....	57.5	41.5	68.0	−14.0	82.0
Mexpetebano 6.....	49.0	34.0	60.0	−19.4	79.4
Mexpetebano 9.....	43.0	29.0	54.0	−25.9	79.9
Mexphalt DX.....	66.0	48.5	76.0	− 8.4	84.4
Mexphalt E.....	57.0	42.5	68.5	−15.7	84.2
Mexphalt E1.....	53.5	37.0	64.0	−16.8	80.8
Sprazuma 5.....	53.5	38.0	64.5	−18.8	83.3
Sprazuma 6.....	55.0	38.0	64.5	−17.1	81.6
Spramex.....	41.0	25.0	51.0	−28.1	79.1
Trinidad Asphalt...	94.0	77.0	104.0	+17.5	86.5
Tar 1.....	3.5	− 6.5	9.0	−38.0	47.0
Tar 2.....	7.9	+ 0.4	14.0	−33.6	47.6
Tar 3.....	30.0	21.0	37.5	−18.7	56.2
Coal-tar Pitch A....	76.0	66.5	83.5	+27.2	56.3
Coal-tar Pitch B....	78.5	67.0	86.8	+28.7	58.1

It is further claimed that the R. & B. and the K. & S. softening-points represent the temperatures at which the substance has attained a *definite* degree of softness, and that these bear a fixed relation to the range in temperature and/or softness between the liquefying- and the solidifying-points, as expressed by the following equation:

$$\frac{\text{Temp. range between softening-point and solidifying-point}}{\text{Temp. range between liquefying-point and solidifying-point}} = C$$

Where C represents a constant, which has been ascertained experimentally to be:

$C_r = 0.8721$ for R. and B. method.
$C_k = 0.6816$ for K. and S. method.

The degree of softness at the liquefying temperature (i.e., liquefying-point) is accordingly fixed at one extreme of an arbitrary scale of softness and assigned a value of 1.0000. Similarly, the degree of softness at the solidifying temperature (i.e., solidifying-point) is fixed at the lower extreme of this scale and assigned a value of 0.0000. Expressed on the same scale, the degree of softness at the R. and B. softening temperature will equal 0.8721 and at the K. and S. softening temperature 0.6816. This relationship is illustrated graphically in Fig. 242.

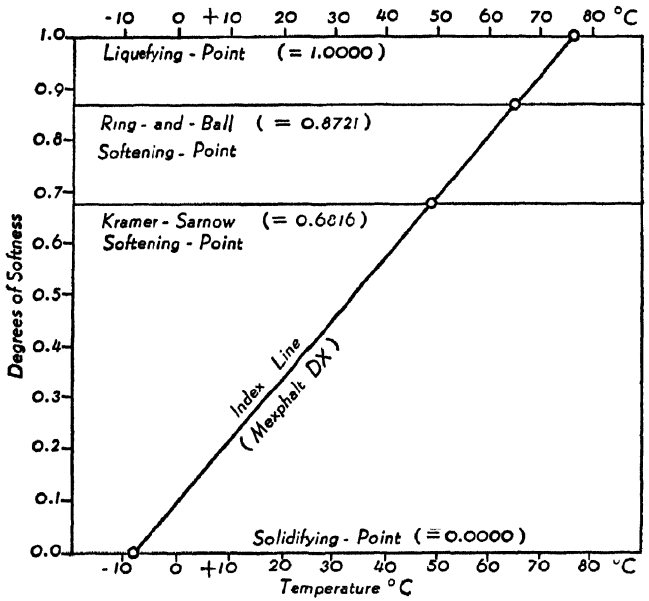


FIG. 242.

Expressed mathematically, these relationships take the following form:

If L = Liquefying-point temperature (Ubbelohde method)
S = Solidifying-point temperature (Metzger method)
R = Softening-point temperature (R. and B. method)
K = Softening-point temperature (K. and S. method)
C_r = 0.8721
C_k = 0.6816

Then:

$$\frac{R - S}{L - S} = C_r = 0.8721$$

and

$$R = 0.8721(L - S) + S \qquad (1)$$

also

$$\frac{K - S}{L - S} = C_k = 0.6816$$

and

$$K = 0.6816(L - S) + S \qquad (2)$$

From (1) and (2) we derive the following:

$$R - 0.9721(L - S) = K - 0.6816(L - S)$$

and

$$R = K + 0.1905(L - S) \qquad (3)$$

$$K = R - 0.1905(L - S) \qquad (4)$$

From the foregoing, it follows that for any bituminous substance, these relationships hold true:

(a) If the liquefying- and solidifying-points are known, then it is possible to calculate either the R. and B. or the K. and S. softening-point (see formulas 1 and 2).

(b) The R. and B. and K. and S. softening-points bear a definite relation to each other, and either one may be computed from the other (see formulas 3 and 4).

These facts have been borne out experimentally with a reasonable degree of accuracy.

Fig. 243 shows the relations between the solidifying-point, the K. and S. softening-point, the R. and B. softening-point, and the liquefying-point of certain of the foregoing substances.

The usefulness of these tests in examining mixtures of bituminous substances will appear from Figs. 244, 245, 246 and 247.

SOFTENING-POINT OR FUSING-POINT*

This constitutes one of the most valuable all-around tests. It is used for purposes of identification, especially with materials fusing at a high temperature, such as the asphaltites, and is particularly useful in this connection upon taking into consideration the specific

* The terms "fusing-point" and "softening-point" have been used throughout the text in place of the phrase "melting-point," since the former are more expressive of the behavior of fusible bituminous substances under the influence of heat. They pass *gradually* from the solid to the liquid condition, the transition taking place slowly, owing to the heterogeneous character of the substances present. The phrase "melting-point" is more appropriately applied to chemical substances having a definite composition, which melt sharply, and within a narrow temperature range.

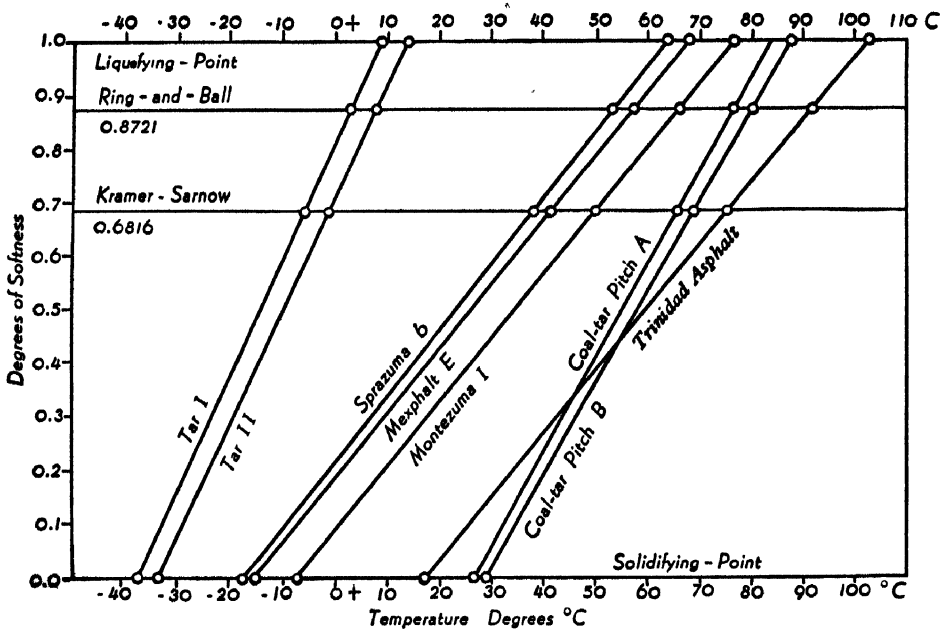


FIG. 243.

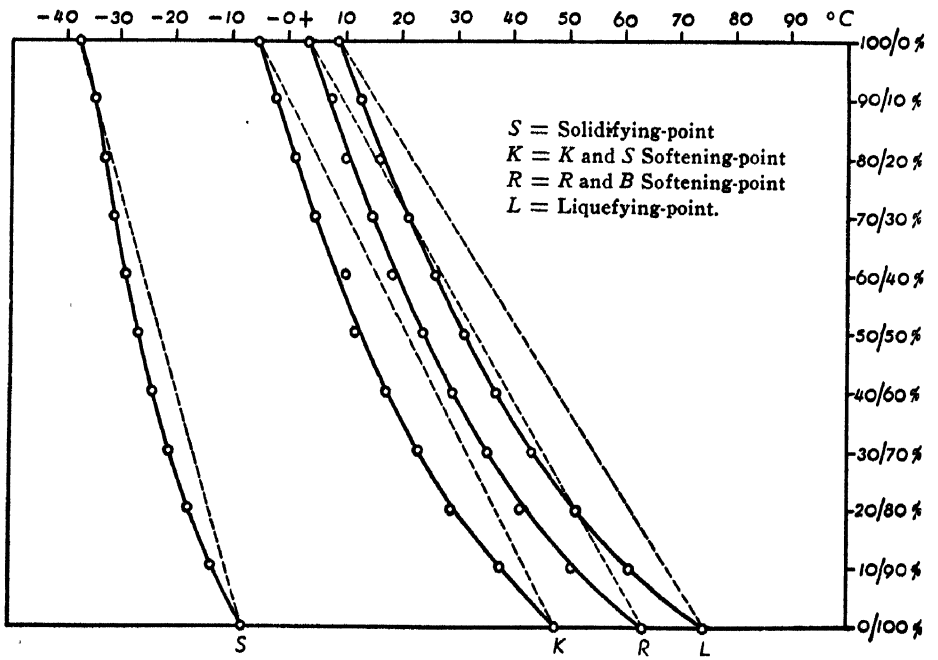


FIG. 244.—Mixtures of Tar I and Mexpetebano.

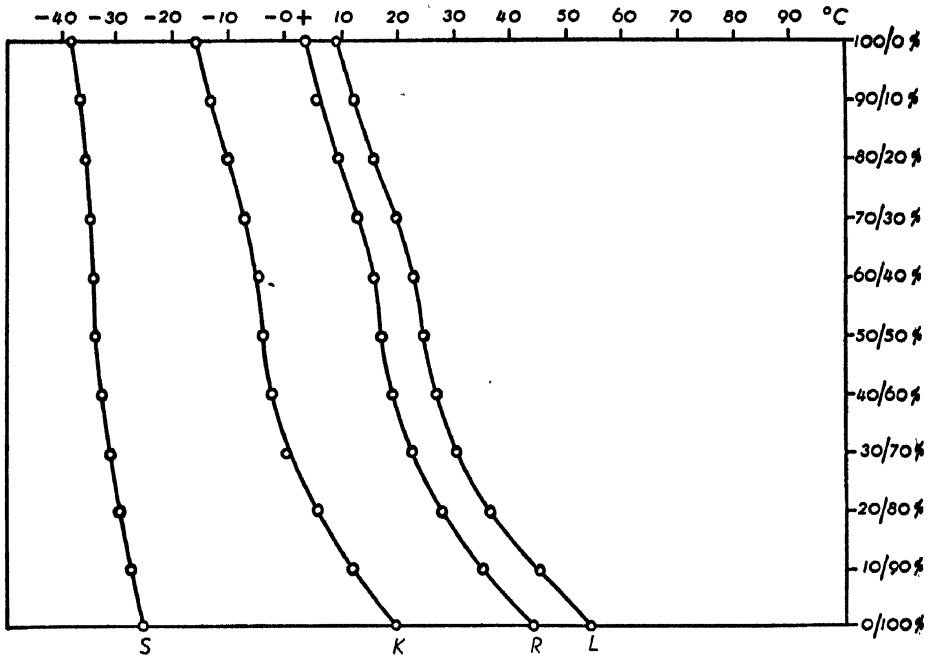


FIG. 245.—Mixtures of Tar I and Mexpetebano 9.

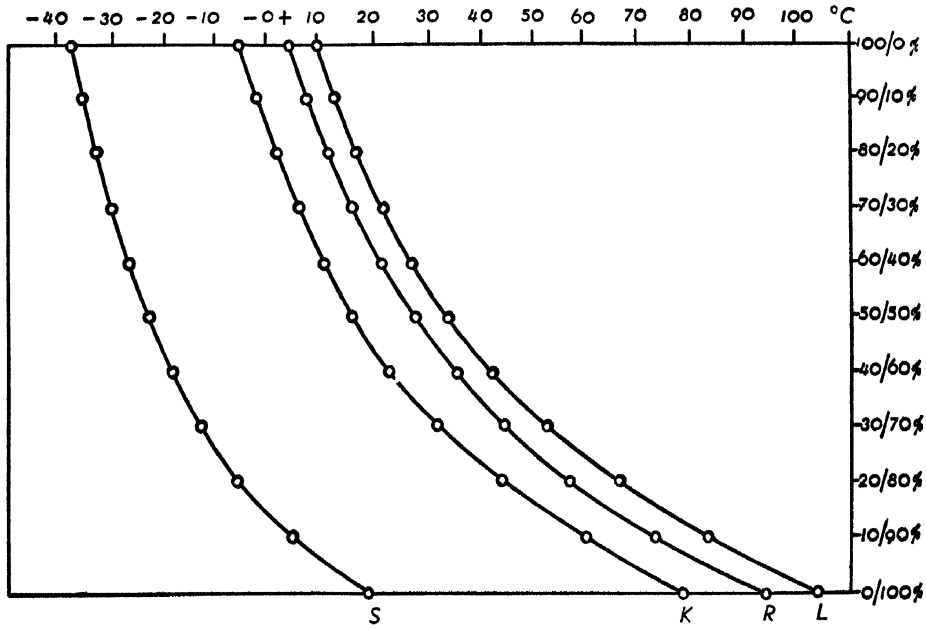


FIG. 246.—Mixtures of Tar 1 and Trinidad Asphalt.

gravity and hardness. It is also used for ascertaining the adaptability of a bituminous material for certain definite usages, including its resistance to the sun or artificial heat. The fusing test serves to gauge the uniformity of supply, and on account of its rapidity and accuracy, is used extensively for purposes of factory control. Various methods have been proposed for this purpose, viz.; change in appearance on heating,[101] heating the powdered substance with sulfuric acid,[102] slide test,[103] sagging tests,[104] dropping tests,[105] extrusion under pressure,[106] releasing a ball under tension,[107] heating the sub-

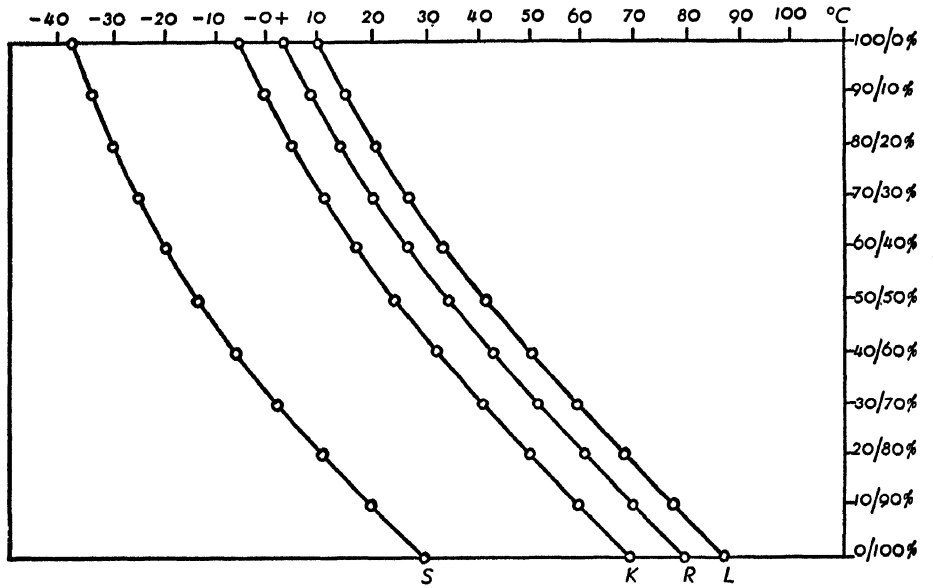


FIG. 247.—Mixtures of Tar 1 and Coal-tar Pitch B.

stance in contact with a thermo-couple and observing the point at which the temperature remains stationary over an appreciable length of time (due to the material absorbing its latent heat of fusion).[108]

Test 15a. Krämer-Sarnow Method. This method is rapid, accurate, and adapts itself either to soft or hard bituminous materials, from residual oils up to grahamite. Its range is greater than that of any other fusing-point method.

It was first proposed by G. Krämer and C. Sarnow.[109] Various modifications have been suggested from time to time.[110] The author, after a careful study of this method, recommends the following procedure:[111]

For Substances Fusing below 176° *F.* This method consists in heating a plug of the bituminous substance 5 mm. long, in an open glass tube, 6–7 mm. internal diameter, and about 8 cm. long, the plug supporting 5 g. mercury, and the tube being immersed in a vessel of water, the level of which reaches approximately the center of the mercury column. In making the test, a thermometer is sus-

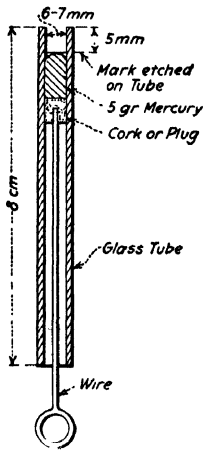


FIG. 248.—Method of Filling K. and S. Fusing-point Tubes.

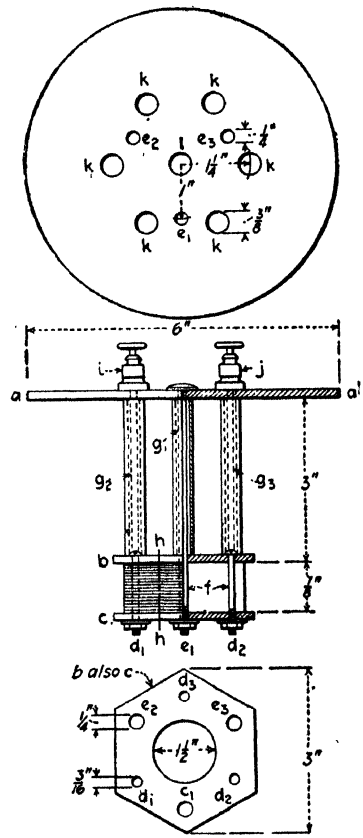


FIG. 249.—Heating Coil for K. and S. Fusing-point Tester.

pended in the liquid, so as its bulb will be at the same level as the plug of bituminous material. The thermometer is supported in a separate glass tube of the same thickness and diameter as the other tube, but differing therefrom in having its lower end sealed, and containing sufficient mercury to surround the bulb. The water is heated at a uniform rate of 4° F. per minute, and the temperature at which the mercury drops through the plug of bituminous material recorded

as its fusing temperature. The tube containing the bituminous substance may have a mark etched 5 mm. from the end, as a convenient guide for the quantity of bituminous material to be introduced. The plug of bituminous material may be introduced into the tube by inverting it and inserting into its lower end a well-fitting cork

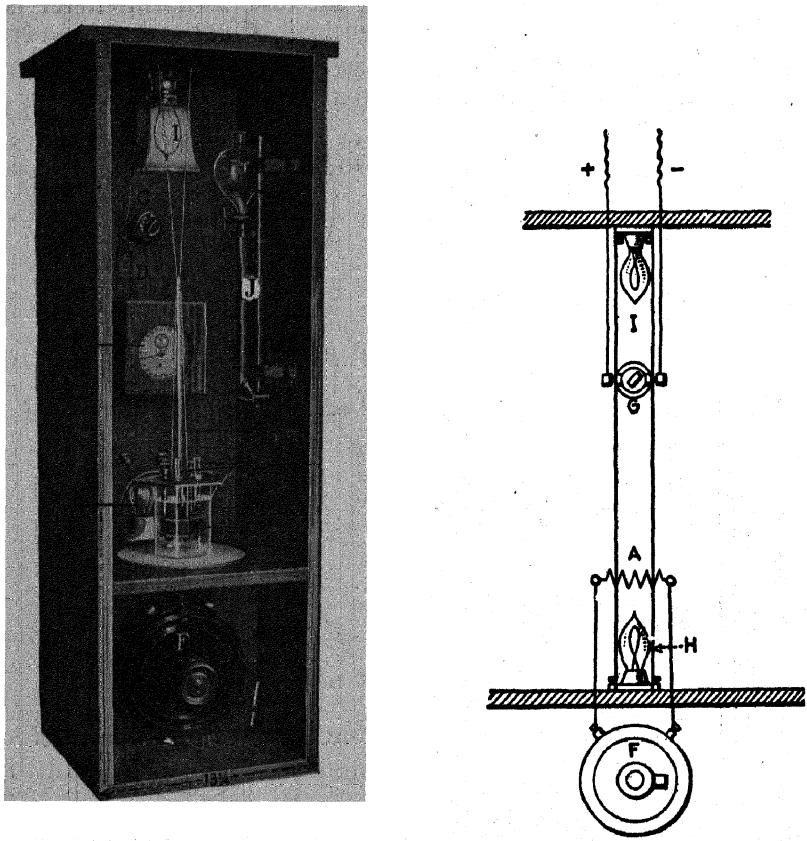


FIG. 250.—K. and S. Fusing-point Tester.

or wooden plug fastened to a stiff wire. The mercury is poured on same, and the plug raised or lowered until the meniscus of the mercury coincides with the mark etched on the tube. The bituminous material is then melted at a temperature slightly above its fusing-point and poured on top of the mercury, to completely fill the tube, which should be warmed slightly. When cool, the bituminous material is levelled off even with the end of the tube, whereupon the tube is inverted and the plug withdrawn. This is illustrated in Fig. 248.

The mercury is measured from a heavy-walled capillary tube of 1 mm. bore, terminating in a three-way cock, as illustrated at *J*, Fig. 250, and calibrated to hold exactly 5 g. mercury at room temperature. The short limb of the tube is connected with a movable reservoir containing mercury, the height of which is adjusted so the mercury in the capillary tube exactly reaches the graduation.

The heating is conveniently effected by an electrical device described by the author,[112] illustrated in Fig. 249, composed of a coil of resistance wire to be immersed in the liquid bath containing the fusing-point tubes, and connected with a rheostat, by means of which the temperature may be controlled to a nicety. Three slabs of slate or asbestos-cement, *a*, *b* and *c*, are fastened together with three small bronze bolts *d*-1, *d*-2 and *d*-3, also three large bolts, *e*-1, *e*-2 and *e*-3 enclosed in glass tubes *f* and *g* respectively, to prevent short-circuiting. The coil *h* consists of 10 yd. of cotton-covered No. 30 nichrome resistance wire wound in a single layer around the tubes, and connected with the bolts *e*-2 and *e*-3, which in turn terminate in binding-posts *i* and *j*. The coil after being assembled is treated with a high-grade insulating varnish and baked until hard. Ten holes are drilled in the slab *a*, three *e*, for the large bolts, six *k* for the fusing-point tubes and one *l*, for the thermometer tube. The coil as described offers a resistance of 75 ohms, and allows a passage of approximately 1.5 amperes at a potential of 110 volts. It will raise the temperature of 500 to 600 ml. of water to the boiling-point in a few minutes, when the full current is applied.

An alternate heating device consists of a resistance cell formed of a beaker 75 mm. diameter by 100 mm. high, carrying 400 ml. water to which are added 4 drops of concentrated sulphuric acid. The apparatus is assembled as shown in Fig. 251. A *direct* current of 110 volts is used in conjunction with a rheostat provided with 25 to 35 notches, having a carrying capacity ranging from ½ up to 100 amperes (corresponding to a resistance of 220 down to 1.1 ohms).[113]

The apparatus is assembled as illustrated in Fig. 250. The heating coil *A* carrying the thermometer *B* and the fusing-point tubes *C* is counterbalanced by the weight *D*, so it may be raised or lowered into the beaker *E* holding 500–600 ml. of water. The heating coil is connected with a rheostat *F* and a switch *G* in parallel with

an 8 c.p. incandescent lamp *H* behind the beaker to illuminate the fusing-point tubes, and a 32 c.p. lamp *I* to light up the interior of the apparatus. *J* represents the mercury measuring-device, and *K* a clock from which the hour hand has been removed, and the dial graduated in 240 divisions representing degrees Fahrenheit. The rise in temperature is synchronized with the minute hand of the clock and controlled by the rheostat to increase *exactly 4° F. per minute*. The initial temperature of the water should be at least 25°

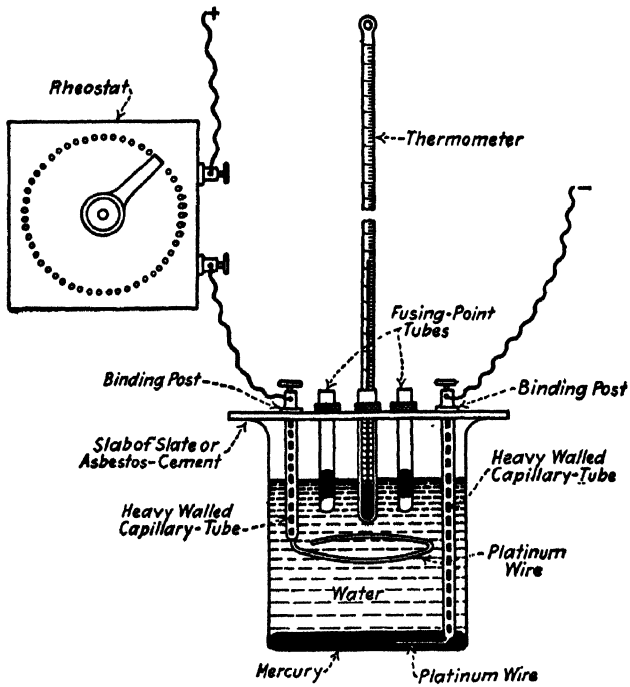


FIG. 251.—Resistance Cell for Fusing-point Determination.

F. lower than the fusing-point of the material to be examined. Six tests may be run simultaneously.

The heating coil is simple to construct, easy to operate, and insures a perfect temperature control. Owing to its skeleton construction, the heat is rapidly dissipated, and there is no danger of the coil burning out, provided it is kept immersed in the water while the current is on. In the author's laboratory, where the coils are in daily use, they last from two to three years, and when burnt out the wiring may be renewed in a few minutes' time.

For Substances Fusing above 176° *F.* In this case the heating is performed by a direct flame, as illustrated in Fig. 252, the water being replaced with castor oil which may be heated safely to about 600° F. This method may be used for determining the fusing-point of asphaltites, including grahamite. A small quantity of the high fusing-point bituminous material is powdered and compressed in the lower end of the fusing-point tube, whereupon it is carefully heated above the flame of a burner, until the plug of bituminous material softens and fuses to the tube, which is evidenced by the color chang-

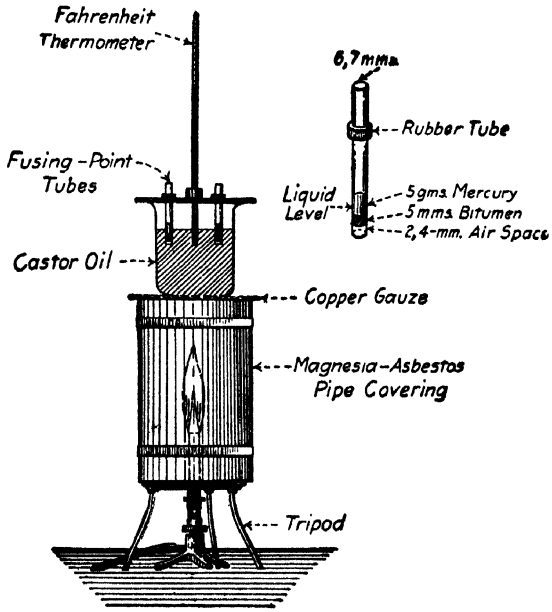


FIG. 252.—K. and S. Tester for High Fusing-point Substances.

ing from a dull to a glossy black. The tube is then stood upright against a block of wood, a snug-fitting glass rod inserted in the upper end, and pressed against the softened bituminous material to compact it into a solid mass 7 to 9 mm. long. On cooling, the plug is then carefully scraped from the lower end of the tube until *exactly* 5 mm. remains, leaving an air space of 2 to 4 mm. between the plug and the lower end of the tube. Care should be taken when suspending the fusing-point tube in the heating bath, to allow the free space below the plug to remain filled with air, otherwise oil will come in contact with and prematurely soften the bituminous material. The bath is heated at the uniform speed of 4° F. per minute.[114]

Test 15b. Ring-and-Ball Method. This has been standardized as follows:[115]

The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow-flowing material to a softer and less viscous liquid. For this reason the determination of the softening-point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

The apparatus shall consist of the following:

(*a*) A brass ring 15.875 mm. (5/8 in.) in inside diameter and 6.35 mm. (1/4 in.) deep; thickness of wall, 2.38 mm. (3/32 in.); permissible variation on inside diameter and thickness of ring 0.25 mm. (0.01 in.). This ring shall be attached in a convenient manner to a No. 13 B. & S. gage brass wire (diameter 1.83 mm. = 0.072 in.). See Fig. 253.

(*b*) A steel ball 9.53 mm. (3/8 in.) in diameter weighing between 3.45 and 3.55 g.

(*c*) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, low form, meets this requirement.)

The sample shall be melted at the lowest possible temperature to avoid loss of volatile constituents[116] and stirred thoroughly, avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling, the excess material shall be cut off cleanly with a slightly heated knife.

For Substances Fusing at 80° *C.* (176° *F.*) *or Below.* Use a thermometer which shall be graduated in either Centigrade or Fahrenheit degrees as may be specified, the ranges being —2 to +80° C. or 30 to 180° F., respectively. Fill the glass vessel to a depth of substantially 8.25 cm. (3.25 in.) with freshly boiled, distilled water at 5° C. (41° F.). Suspend the ring containing the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper surface is 5.08 cm. (2 in.) below the surface of water. Place the ball in the water but

not on the specimen. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm. (¼ in.), but not touching the ring. Maintain the temperature of the water at 5° C. (41° F.) for fifteen minutes.* With suitable forceps, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly as in Fig. 253. Apply the heat in such a manner that the temperature of the water is raised 5° C. (9° F.) each minute. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening-point. No correction shall be made for emergent stem. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be ±0.5° C. (0.9° F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

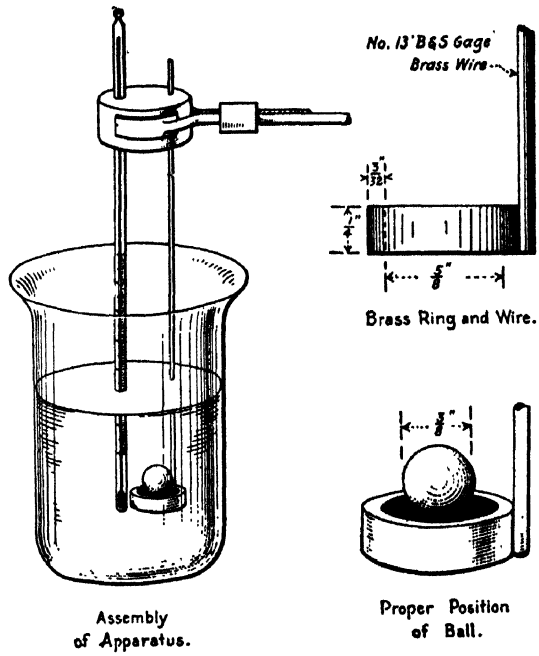


FIG. 253.—Apparatus for Ring-and-Ball Method.

For Substances Fusing above 80° C. (176° F.). The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 30 to 160° C., or 85 to 320° F., respectively. The same method as given above shall be employed, except that U. S. P. glycerin shall be used instead of water, and the starting point of the glycerin bath shall be 32° C. (89.6° F.). The bath shall be brought to this temperature and thoroughly agitated, then the apparatus and specimens shall be placed in the bath which shall be maintained under agitation

* NOTE BY AUTHOR.—It will be found more convenient, and the accuracy of test will in no way be sacrificed, if the initial temperature of the water is maintained 25° F. below the softening point of the substance under examination.

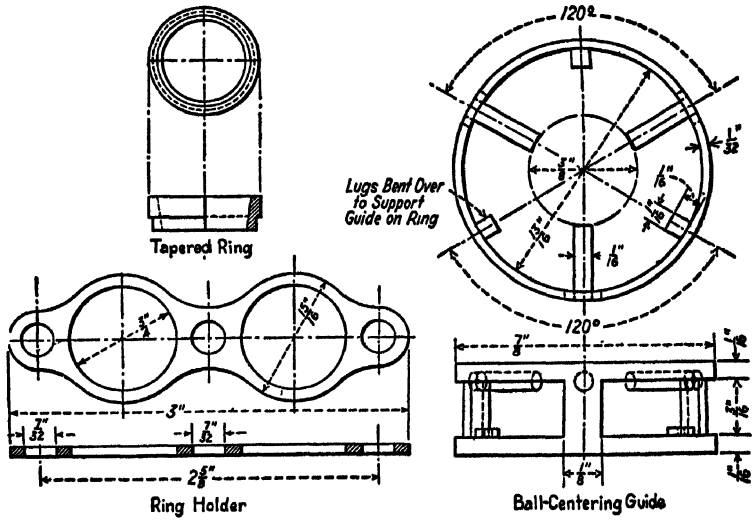
at the starting temperature for fifteen minutes, after which the assembly shall be completed by placing the ball on the center of the specimen and the test carried on as above. In applying the heat, the ring apparatus shall be placed off the center of the container and the burner placed midway between the center and edge of the beaker away from the specimen.

Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results. A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning. The limit of accuracy of the test is ±0.5° C. (0.9° F.).

To adapt the method for testing substances which may shrink away from the cylindrical ring, as for example, resins, waxes, etc., a tapered brass ring of the following dimensions has been proposed:[117] depth: 6.35 ± 0.10 mm. (¼ in.); inside diameter at bottom: 15.88 ± 0.10 mm. (⅝ in.); inside diameter at top: not more than 17.6 mm. and preferably 17.46 mm. (11/16 in.); outside diameter at bottom: not less than 18.0 mm. nor more than 20.6 mm.; outside diameter at top: not less than outside diameter at bottom nor more than 20.6 mm. ± 0.10 mm. A ball-guide for centering the balls, constructed of brass, and having the shape and dimensions illustrated in Fig. 253A is recommended.

The sample shall be selected from the material to be tested in such a manner as will avoid the inclusion of the surface layer and a quantity at least twice that necessary to fill the desired number of rings and in case less than 40 g. shall be melted immediately in a clean container, using an oven, sand or oil bath to prevent local overheating. Care shall be taken to avoid incorporating air bubbles in the sample which should not be heated more than 30° C. (54° F.) above its softening point, in the case of materials having a softening point below 121° C. (250° F.), except rosin, or more than 50° C (90° F.) for rosin and materials with softening points above 121° C. (250° F.); for materials having a long softening range it may be necessary to increase this to 85° C. (185° F.), the time from the beginning of heating to the pouring of the sample not to exceed 15 min. Immediately before filling the rings, they shall be preheated to approximately the temperature at which the ma-

terial is to be poured. The rings, while being filled, should rest on an amalgamated brass plate. The quantity of material poured into the rings shall be such that after 20-min. cooling at room temperature of materials with softening points below 80° C. (176° F.) and 40-min. cooling for materials of higher softening point, an excess amount will remain which shall then be cut off cleanly with a slightly heated spatula. For routine testing this period of time may be curtailed according to the characteristics of the material



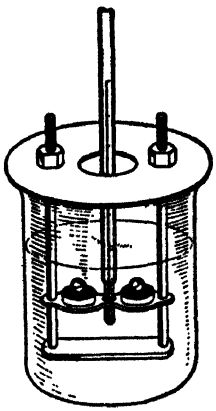
Courtesy A.S.T.M.

FIG. 253*a*.—Tapered Ring, Ball Centering Guide and Ring Holder.

under test. In case the test is to be repeated, a clean container and fresh sample shall be used.

(*a*) *Assembling Apparatus.*—The glass container shall be filled to a depth not less than 9 cm. (3.5 in.) with freshly boiled distilled water (or with glycerin for materials melting above 80° C. (176° F.)) which has been cooled to not less than 45° C. (81° F.) below the anticipated softening point but in no case lower than 5° C. (41° F.) for materials melting below 80° C. (176° F.) and in no case lower than 35° C. (95° F.) for materials melting above 80° C. (176° F.). The ring containing the sample and ball guide shall be suspended in the water so that the lower surface of the filled ring is 2.5 cm. (1 in.) above the upper surface of the lower horizontal plate which is at least 1.3 cm. (½ in.) above the bottom of

the glass vessel (Fig. 253*b*) and so that its upper surface is at least 5.1 cm. (2 in.) below the surface of the water. The ball shall be placed in the water but not on the specimen. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. (⅜ in.) but not touching the ring. The initial temperature shall be maintained for 15 min. With suitable forceps the ball shall be placed in the center of the upper surface of the material in the ring, using the ball guide to hold the ball in this position.



Courtesy A.S.T.M.

FIG. 253*b*.—Assembly of Apparatus Showing Two Rings

(*b*) *Heating.*—To facilitate uniform heating, the burner shall be placed midway between the center and the edge of the beaker on a diameter at right angles to the diameter which includes the rings and the thermometer bulb. The effects of drafts on uniformity of heating must be eliminated, using shields if necessary. The heat shall be applied in such manner that the temperature of the bath is raised 5° C. (9° F.) each minute.

The temperature reading to one-half of the smallest scale division indicated by the thermometer at the instant the sample touches the bottom of the container (or the lower horizontal plate) shall be taken as the softening point.

The electrical heating coil described in Test 15*a* may be used to good advantage in the Ring-and-Ball method, but the length of nichrome wire should be reduced to 6 yd., to provide for the more rapid heating of the bath, or the resistance-cell illustrated in Fig. 251 may be used as an alternative.

Tests made by the author indicate that the Ring-and-Ball fusing-points range 15 to 25° F. higher than those obtained by the K. and S. method, although it is questionable whether a fixed mathematical formula adapts itself to the relationship, on account of the basically different fundamentals involved in the two methods of test.[118] Proctor has made a comparison of the K. and S., R. and B., the flowing-temperature and other fusing-point tests.[119]

Test 15c. Cube Method. This method is restricted to testing tar-pitches. The following procedure has been proposed:[120]

The softening of pitch takes place at no definite moment or temperature. As the temperature rises, pitch gradually and imperceptibly changes from a brittle or exceedingly thick and slow-flowing material to a softer and less viscous liquid. For this reason the determination of the softening-point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

The apparatus shall consist of the following:

(*a*) A mold suitable for forming a 12.7-mm. (½-in.) cube of pitch. A recommended type is shown in Fig. 254.

(*b*) An L-shaped right-angled hook made of No. 12 B. and S. gage copper wire (diameter 2.05 mm. =0.0808 in.) the foot of which shall be 2.54 cm. (1 in.) long. See Fig. 254.

(*c*) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, Griffin low form, meets this requirement.)

(*d*) A thermometer which shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being —2 to +80° C. or +30 to +180° F., respectively.

The pitch shall be formed into a 12.7-mm. (½-in.) cube, truly shaped and with sharp edges, either by melting and pouring, or softening and pressing, into a mold. In all cases an excess of pitch shall be used and the surplus material shall be cut off cleanly with a slightly heated knife. The harder pitches specified can ordinarily be molded at room temperature, the softer pitches in water at about 4° C. (39.2° F.). If they are melted, they should first be thoroughly stirred, avoiding incorporating air bubbles in the mass, and then poured into the mold so as to leave an excess on cooling. The mold should rest on a brass plate and the surface of the plate and the interior surfaces of the mold should be amalgamated to prevent the pitch from adhering to them.

For Pitches Fusing between 110 *and* 170° *F.* Assemble the apparatus as shown in Fig. 254. Fill the glass vessel to a depth of substantially 9.5 cm. (3.75 in.) with freshly boiled, distilled water at 15.5° C. (60° F.). Place the cube of pitch on the wire as shown in Fig. 254 and suspend it in the water so that its lower edge is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its

upper edge is 5.08 cm. (2 in.) below the surface of the water. Allow it to remain in the water for fifteen minutes before applying heat. Suspend the thermometer so that the bottom of the bulb is level with the bottom edge of the cube of pitch and within 0.635 cm. (¼ in.), but not touching the cube. Apply the heat in such a manner that the temperature of the water is raised 5° C. (9° F.)

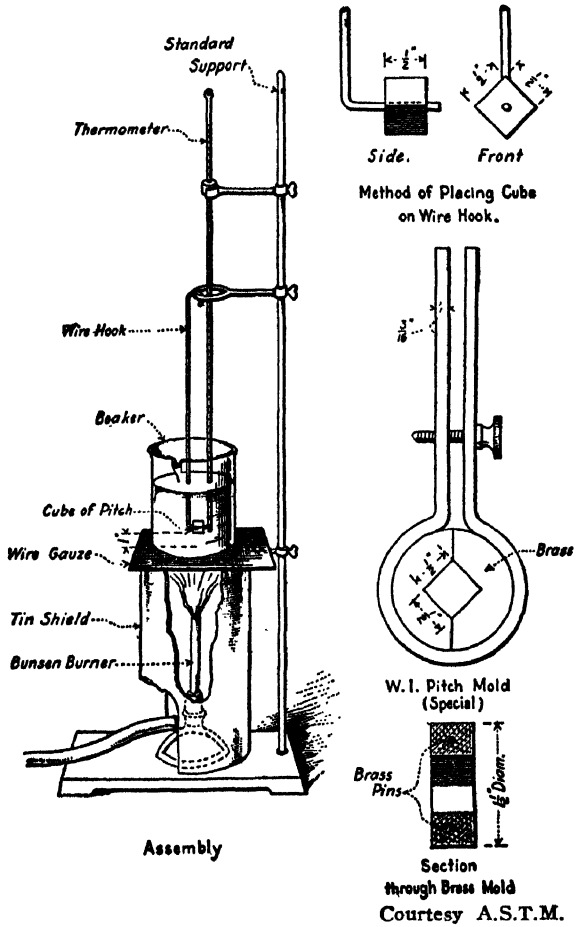


FIG. 254.—Apparatus for Cube-in-Water Method.

each minute. The temperature recorded by the thermometer at the instant the pitch touches the bottom of the glass vessel shall be reported as the softening-point. No correction shall be made for emergent stem. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first

three shall be ±0.5° C. (0.9° F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

For Pitches Fusing below 110° *F.* Use the same method as given above, except that the water when placed in the glass vessel shall be at a temperature of 4° C. (39.2° F.). The cube shall be allowed to remain fifteen minutes in this water before applying the heat.

The use of freshly distilled water is essential, as otherwise air bubbles may form on the cube and retard its sinking. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results. A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the pitch from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning. The limit of accuracy of the test is ±0.5° C. (0.9° F.).

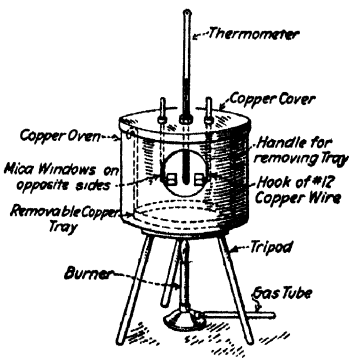


FIG. 255.—Cube-in-Air Method for High Fusing-point Substances.

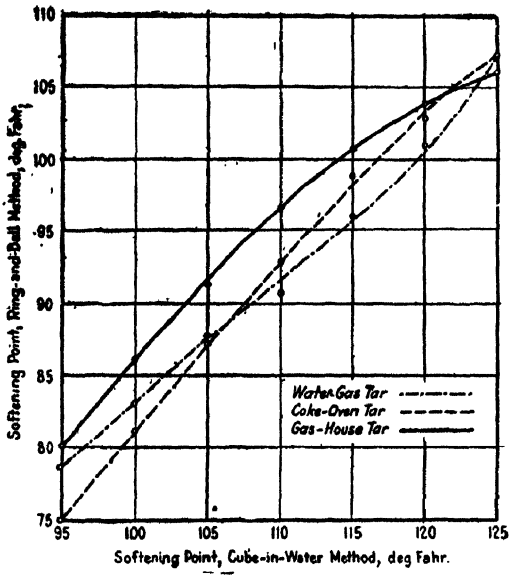
For Pitches Fusing above 170° *F.* The heating is performed in an air bath in the apparatus illustrated in Fig. 255. The cube should be suspended in line with the observation windows, and the thermometer bulb brought to the same level. The temperature is raised 9° F. per minute, and recorded by the thermometer when the cube drops 1 in.

Investigations of the relationship between the Cube and the Ring-and-Ball methods [121] indicate that the results vary considerably, depending largely upon the nature of the products tested and their fusing-points. No exact factors can be given. The relation between the fusing-point by the Cube method and the results obtained by the Schutte consistency tester, the Engler viscosimeter, also the float test have been investigated.[122]

Figure 256 shows the relation between the softening-points obtained by the Cube-in-Water and the Ring-and-Ball methods on three types of tars, each of which were evaporated and samples taken having softening points by the Cube-in-Water method of 95, 100, 105, 110, 115, 120 and 125° F., respectively.[123]

Test 15d. Compression Method. By this test the extent of softening is measured progressively as the temperature is increased, by a specially constructed instrument illustrated in Fig. 257, known as the Widney Resiliometer.[124] The behavior of the material is expressed graphically and forms a clear picture of the effect of heat on its physical condition.

It consists of a large dial *A* graduated in 100 divisions, each representing .001 in. movement of the plunger *B*, so that one revolution of the pointer, measuring 1/8 in. in diameter, corresponds to 0.1 in. travel. A small supplemental dial *C* registers the number of revolutions of the pointer on the large dial *A*. By means of a mold, a specimen is prepared measuring exactly 0.1 in. thick and about 2 1/2 × 1/2 in. in area. After allowing the specimen to stand at least twelve hours before testing, it is placed on the base *D*, the plunger *B* brought in contact, and the reading observed on the dial *A*. The catch *E* is then pressed which releases the weight *F* attached to the quadrant *G*. This, by means of the rack *H* and the pinion *I*, transmits the pressure to the plunger *B*. The weight *F* is approximately 2 1/2 lb., so that the material will be subjected to a pressure equivalent to 200 lb. per sq. in. The pressure is maintained until the substance reaches the limit of compression, whereupon the reading on the dial is taken and compared with the original thickness. For example, a specimen showing a thickness originally of 100 mils and giving a reading of 25 mils, would be reported to show a compression of 25 per cent. The specimen is heated at the rate of 4° F. per minute and tested every 10° F. at different points over its surface. The diagram in Fig. 258 shows the results obtained upon



Courtesy A.S.T.M.

FIG. 256.—Comparison of Ring-and-Ball and Cube-in-Water Softening Points for Different Types of Tars.

testing various commercial bituminous substances in the foregoing manner.

Sundry other methods have been suggested to measure the temperature at which a needle or rod under a given load will penetrate the bituminous substance through a predetermined distance; likewise a method involving the measurement of the indentation of a loaded metal sphere acting for a given time at a predetermined temperature.[125]

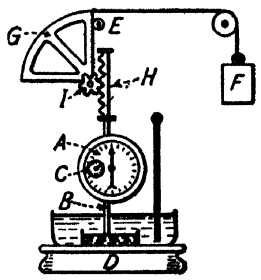


FIG. 257. — Resiliometer for Measuring Compression.

Test 15e. A.S.T.M. Method for Petrolatum. The following procedure has been adopted as standard.[126]

The "A.S.T.M. Petrolatum Melting-point" represents the temperature at which petroleum becomes sufficiently fluid to drop from the thermometer used in making the determination under definite prescribed conditions.

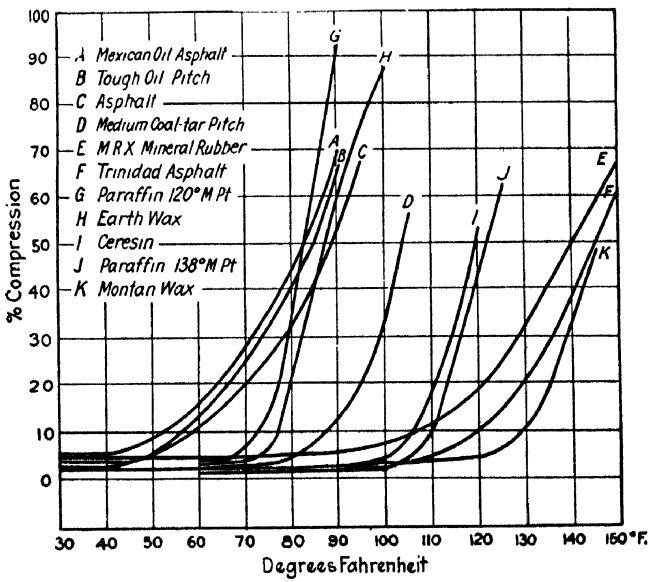


FIG. 258.—Results Obtained with the Resiliometer.

The melting-point thermometer shall conform to the specifications for the A.S.T.M. paraffin-wax melting-point thermometer. The bath thermometer shall be of any suitable type, accurate throughout the required range to 2° F.

The test tubes shall be 25 by 100 mm. in size, and shall be supplied with corks grooved at sides to permit air circulation and bored at the exact center to receive the thermometer. A transparent container of not less than 500-ml. capacity that will permit the immersion of the test tube to a depth of at least 75 mm. and still leave a depth of 15 mm. of water below the bottom of the test tube.

Samples of sufficient size that exactly represent the petrolatum under inspection shall be secured. The material shall be melted slowly in a casserole or other suitable dish with stirring until the temperature reaches 200° F. A fresh portion of the sample shall be used for each determination. The source of heat shall then be removed and the petrolatum allowed to cool to 15° F. above the temperature that it is anticipated will be its melting-point. The thermometer bulb shall be chilled to 40° F., wiped dry and while still cold thrust into the melted petrolatum so that approximately the lower half is submerged. It shall be withdrawn immediately, held vertically away from the heat until the surface dulls, and then placed in a water bath having a temperature not over 60° F. for five minutes.

The thermometer shall be securely fixed in a test tube by means of a cork so that the lowest point is 15 mm. above the bottom of the test tube. The test tube shall be surrounded with a water bath at a temperature of 60° F. The temperature of the bath shall be raised at a rate of 3° F. per minute to 100° F., then at a rate of 2° F. per minute until the first drop of petrolatum leaves the thermometer. The temperature at this instant shall be recorded.

If the variation of three such determinations does not exceed 2° F., the average of these three determinations shall be reported as the melting-point of the sample under test. If the variation of three determinations is greater than 2° F., two additional determinations shall be made and the average of five determinations reported as the melting-point.

Test 15f. A.S.T.M. Method for Paraffin Wax. The following method has been standardized: [127]

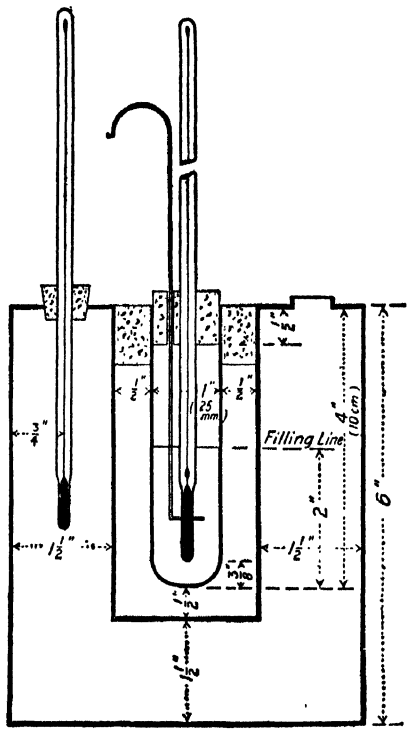
The "Paraffin-Wax Melting-point" represents the temperature at which melted paraffin wax, when allowed to cool under definite prescribed conditions, first shows a minimum rate of temperature change.

NOTE.—The so-called "American Melting-point" is an arbitrary figure 3° F. higher than the A.S.T.M. Paraffin-Wax Melting-point.

(*a*) Wax Container: Test tube of standard form, 25 mm. (1 in.) outside diameter and 100 mm. (4 in.) long. It may be marked with a filling line, 2 in. above the bottom. This test tube shall be closed in a tightly fitting cork having two openings, one at the center for the melting-point thermometer and the other for a stirrer at one side of the center. The opening for the stirrer may be lined with glass or metal tubing to act as a guide for the stirrer.

(*b*) Air bath: Suitable water-tight cylinder, 2 in. in inside diameter and 4½ in. deep. This air bath shall be provided with a tightly fitting cork having a central opening for holding the test tube firmly in a vertical position in the center of the air bath.

(*c*) Water Bath: Suitable cylinder, 5⅛ in. in inside diameter and 6 in. deep. This water bath shall be provided with a suitable cover and with the guides and fasteners necessary to hold the air bath firmly in a vertical central position, so that the sides and bottom of the air bath shall be surrounded by a layer of water 1½ in. thick. The water-bath cover shall have a slot for introduction of a suitable stirrer and shall have an opening for the bath thermometer, so that the latter may be suspended in a vertical position ¾ in. from the outside wall of the water bath. Air bath, water bath and water-bath cover may be conveniently made of metal in one assembly as shown in Fig. 259.



Courtesy A.S.T.M.

FIG. 259.—Apparatus for Determination of Melting Point of Paraffin Wax.

(*d*) Stirrer in Test Tube: Brass or copper wire, ⅛ in. in diameter and about 12 in. long. A circular loop, ½ in. in diameter, shall be formed at one end of this wire in such a manner that the

loop lies in a horizontal plane when the rest of the wire is in a vertical position. The stirrer thus formed shall be passed through the proper opening in the test-tube cork and the upper end may then be bent into a shape convenient for holding.

(*e*) Thermometers: The Paraffin-Wax Melting-point thermometer shall be a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 27 to 71° C. or 80 to 160° F., respectively. Bath thermometers shall be of any suitable type, accurate to 2° F. throughout the required range.

An average sample of the wax to be tested shall be melted in a suitable container in a water bath whose temperature shall be not more than 35° F. above the approximate melting-point of the wax sample. Direct heat, such as a flame or hot plate, shall not be used and the wax sample shall not be held in the melted condition any longer than necessary.

The test tube shall be filled with melted wax to a height of 2 in. The test-tube cork, carrying the stirrer and the melting-point thermometer with the 3⅛-in. immersion line at the under surface of the cork, shall be inserted into the test tube for a distance of ½ in. The lower end of the thermometer bulb shall then be ⅜ in. from the bottom of the test tube. The air bath being in its proper position in the water bath, the latter shall be filled to within ½ in. of the top with water at a temperature 15 to 20° F. below the approximate melting-point of the wax sample.

The test tube containing the melted wax, with wax stirrer and thermometer in place, shall be inserted into the air bath in a central vertical position so that the bottom of the test tube is ½ in. from the bottom of the air bath. The temperature of the water bath shall be adjusted by stirring if necessary, so that it shall be lower than the temperature of the wax sample by not more than 30° F. and not less that 25° F., when the wax sample has cooled to a temperature 10° F. above its approximate melting-point.

When these conditions have been obtained, temperature adjustment and stirring of the water bath shall be discontinued. The wax shall be stirred continuously during the remainder of the test, the stirring loop being moved up and down throughout the entire length of the test tube in a steady motion at the rate of 20 complete cycles

per minute. The melting-point thermometer reading, estimated to 0.1° F. shall be observed and recorded every thirty seconds. The temperature of the wax will fall gradually at first, will then become almost constant and will then again fall gradually.

The melting-point thermometer reading, estimated to 0.1° F., shall be observed and recorded every thirty seconds, for at least three minutes when the temperature again begins to fall after remaining almost constant. The record of temperature reading shall then be inspected and the average of the first four readings that lie within a range of 0.2° F. shall be considered as the uncorrected melting-point. This temperature shall be corrected if necessary for error in the thermometer scale and the corrected temperature shall be reported as the "Paraffin-Wax Melting-point." Duplicate determinations on the same sample should differ by not more than 0.2° F.

A method has been proposed for determining the melting-point of paraffin-wax micro-analytically, where but small quantities are available for test.[128]

FLOW-POINT

Test 15g. Richardson's Method. This test [129] consists in forming in a suitable mold, cylinders ¾-in. long and ⅜-in. in diameter of the substance, placing them on a brass plate with corrugations corresponding in size to that of the cylinders, and exposing same at an angle of 45° to the desired temperature in an air-oven for a predetermined length of time. The brass flow-plate is 8 by 2⅜ in. in size and is provided with four corrugations. The distance which the specimens flow is measured at the conclusion of the test and represents the extent of flow. A modification of this test has been adapted for testing Bituminous Enamel for the inside of steel ships (Chapter XXVII).

LIQUEFYING-POINT

Test 15h. Ubbelohde's Method. This test has been standardized in Germany [130] and represents the temperature at which the substance will fall in the form of a drop under its own weight under prescribed conditions. The apparatus is shown in Fig. 260, and consists of a thermometer "a" provided with a bulb 6 ± 0.6 mm. long and 3.5 ± 0.4 mm. in diameter, to the lower end of which is fastened a metal collar "b," provided with a vent "c." A small

copper thimble "e," illustrated dimensionally in "A," 10 mm. long, having an opening at its lower end 2 mm. long and 3 mm. (± 0.05) in diameter, is filled flush to the top with the softened bituminous substance, and then inserted in the collar until it engages the three prongs "d," whereupon the thermometer-bulb should assume a position exactly 2.5 mm. above the upper rim of the opening in the thimble "e." This may be controlled by previously checking with a metal mandrel, illustrated dimensionally in "B." By means of a cork, the thermometer is suspended in a glass test-tube 200 mm. long, 40 mm. outside diameter, having walls 0.75 mm. thick, which in turn is immersed for 2/3rds of its length in a beaker (185 mm. high, 90 mm. outside diameter at its lower end, having walls 1 to 1.5 mm. thick) filled with water. The specimen is allowed to cool until the thermometer registers room temperature, whereupon the beaker is heated with a burner so that the temperature registers a rise of exactly 1° C. per minute. The "liquefying-point" or "dropping-point" represents the temperature at which the first drop of the substance falls from the lower end of the copper thimble "e." Glycerin or castor oil may be substituted for the water, where the substance has a "liquefying-point" above 80° C.

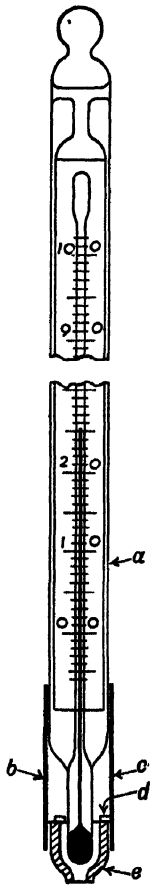


FIG. 260.—Ubbelohde's Liquefying-point Tester.

TWISTING-POINT

Test 15i. Taylor's Method. This test has been proposed by H. F. Taylor [181] and is of value in ascertaining the temperature at which the substance may be twisted under torsion without fracturing. The apparatus is illustrated in Fig. 261, being constructed in brass, and consists of a device for the application of a standard torque to a square prism of the substance molded in a section exactly 75 by 13 by 13 mm. The torque is exerted by a weight of 150 g. suspended by a thread, which after passing over a pulley, is attached to a drum, the circumference of which is equal in dimension to the perimeter of a cross-section of the test specimen, namely

52 mm. The drum is fixed to a vertical spindle free to rotate, the lower end of which is attached to a square socket 15 mm. deep. A

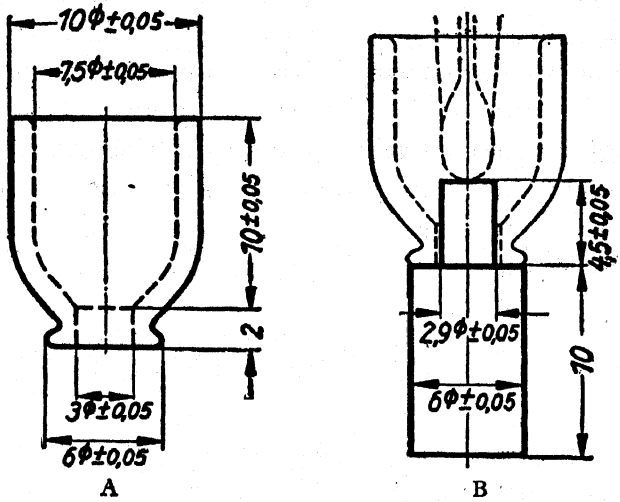


FIG. 260.—Details of Liquefying-Point Tester.

second stationary socket, also 15 mm. deep is attached to the lower framework. The distance between the two sockets is exactly 50 mm. The test specimen is inserted in the sockets and the pointer set at the zero mark on the scale. The beaker is filled with water at 20° C. to a point at least 50 mm. above the upper end of the specimen, and after being allowed to stand for at least 10 minutes, the weight is attached to the hook, and the water heated at the uniform rate of 2° C. per minute.

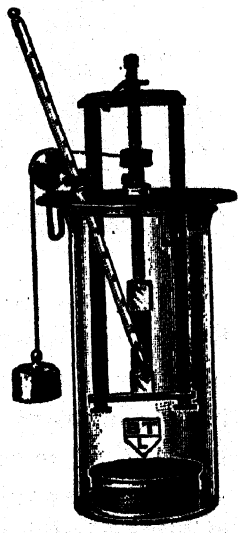


FIG. 261.—Twisting-point Tester.

The temperature is recorded when the specimen has twisted through an arc of exactly 180 deg. and is reported as the "twist-point" of the material under examination. This same instrument may be used for determining the resilience of the material, as follows: the test is repeated on another specimen and the water maintained at the temperature of the "twist-point" for twenty minutes before any weight is attached. The pointer is set at zero, and a 500-g. weight suspended. When the specimen has twisted through an arc of exactly 360 deg., the weight is sud-

denly lifted by hand and the recoil noted. When equilibrium has been reached, the reading is taken, and the number of degrees' recoil is recorded as the "resilience" or "elasticity" of the material.

This same apparatus may be used for ascertaining the absolute viscosity of the substance, in which case the sample is permitted to twist through two complete revolutions before readings are taken, after which the next two or three complete revolutions are timed with a stop watch and the average time per revolution noted at any predetermined temperature. The viscosity may be calculated from the following formula:

$$V = \frac{mgrlt}{1000\,\pi^2 R^4}$$

where V = viscosity in kilopoises; m = weight of torque (i.e., 50 g.); g = acceleration due to gravity (981 g./sec.^{2}); r = radius of torque-drum plus cord; l = length of sample in mm.; t = time of revolution in seconds; and R = radius of sample.

VOLATILE MATTER

This test is used for identifying various bituminous materials. Thus in the case of asphalts, the volatilization test will often serve to identify soft native asphalts, which contain larger percentages of volatile matter than soft residual or blown petroleum asphalts. Cut-back products also carry a large percentage of volatile constituents.

The test may also be used to determine the adaptability of a bituminous substance for certain definite purposes, where it becomes necessary to heat it to high temperatures, as for example in the paving industry or in manufacturing bituminized roofings and floorings. It likewise serves as a valuable adjunct for gauging the uniformity of supply and for purposes of factory control. It also furnishes an *indication* of the weather-proof properties of the material. Other things being equal, bituminous substances showing the smallest percentage of volatile matter will prove most weatherproof on exposure to the elements. It should be noted, however, that the volatility test alone must not be taken as the final criterion as to

whether or not a bituminous substance is weatherproof, since other factors should also be taken into consideration. The volatility test may be regarded as an accelerated test, showing the loss of volatile constituents exclusive of water which will take place upon exposure to the weather in a relatively thin layer, for a long time.

Test 16. A.S.T.M. Method. The following method has been adopted as standard.[132]

This test covers the determination of the loss in weight (exclu-

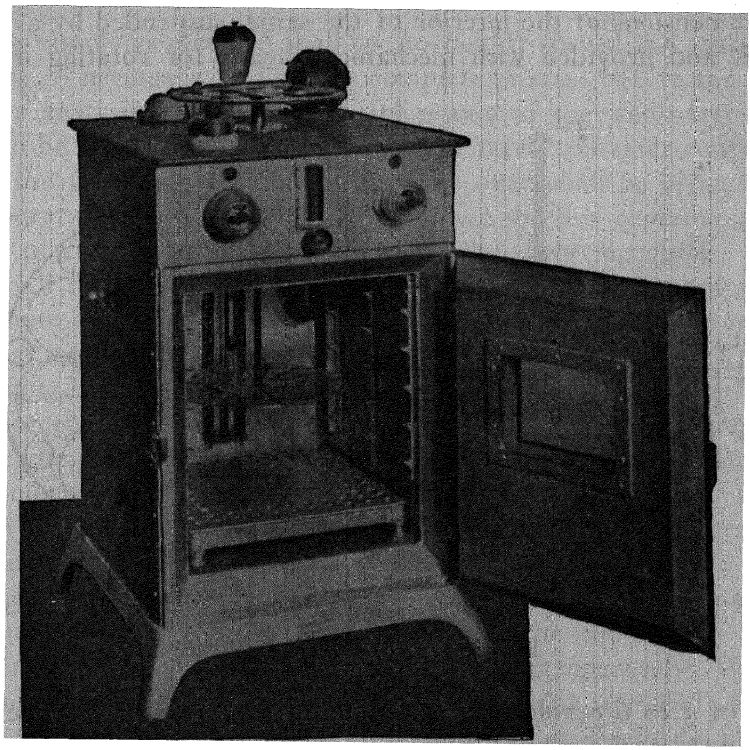


FIG. 262.—Electrically-heated Volatility Oven.

sive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination shall, therefore, first be tested for water and if water is found to be present, it shall be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample shall be obtained which is free from water.

The oven shall be rectangular in form with double walls and

heated by electricity, as illustrated in Fig. 262. Its interior dimensions shall be as follows: height, exclusive of space occupied by the heating element, not less than 29.26 cm. (11.5 in.), width and depth, each at least 5.08 cm. (2 in.) greater than the diameter of the revolving shelf. The oven shall be provided with a perforated metal circular shelf approximately 24.8 cm. (9.75 in.) in diameter. A recommended form of aluminium shelf is shown in Fig. 263. This shelf shall be placed in the center of the oven, with respect to all dimensions of the interior of the same, suspended by a vertical shaft and provided with mechanical means for rotating it at the

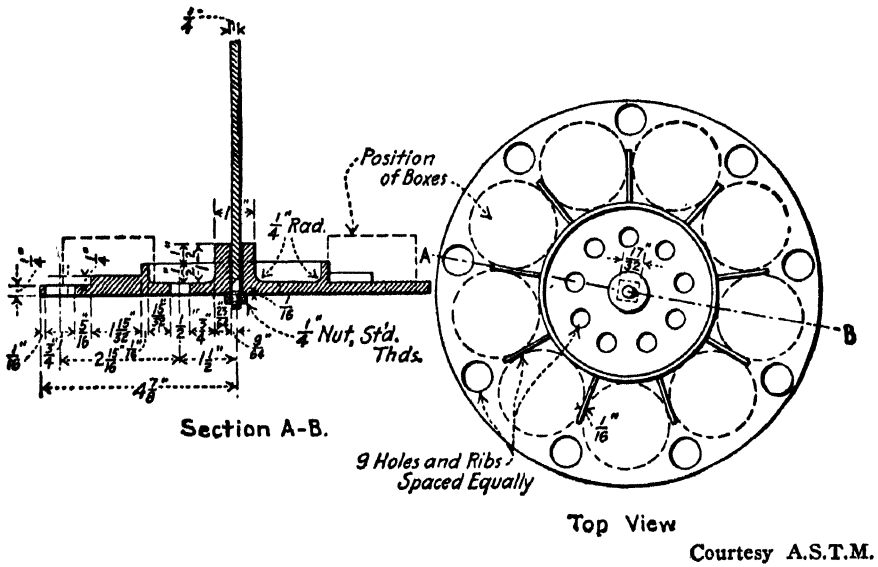


FIG. 263.—Shelf of Volatility Oven.

rate of 5 to 6 revolutions per minute. One side of the oven shall be hinged and equipped to serve as a tight-fitting door which shall contain a window at least 4 in. square, with double glass, through which a thermometer, located in front of and level with the revolving shelf, may be read without opening the door. One round vent hole shall either be placed in the top and bottom of the oven, or one only near the top, and one only near the bottom at each side of the oven. Each hole shall be not less than 1.27 cm. (0.5 in.) nor more than 1.60 cm. (0.63 in.) in diameter and they shall both be left open during the operation of the oven.

The thermometer shall be graduated in Centigrade degrees, the range being 155 to 170° C.

The container in which the sample is to be tested shall be of metal or glass, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows: diameter, 55 mm. (2.17 in.); depth, 35 mm. (1.38 in.).

NOTE.—The American Can Co.'s 3-oz. Gill style flat-bottom, seamless ointment box, deep pattern, fulfills these requirements.

The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed. Fifty grams of the water-free material to be tested shall be weighed into a tared container conforming to the foregoing requirements. The oven shall be brought to a temperature of 163° C. (325° F.), and the box containing the sample placed in one of the recesses of the revolving shelf. The oven shall then be closed and the shelf rotated 5 to 6 revolutions per minute during the entire test. The temperature shall be maintained at 163° C. ± 1° C. (325° F. ± 1.8° F.) for 5 hours after the sample has been introduced and the oven has again reached that temperature. The sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated. During the 5-hour period the temperature shall not vary more than ± 1° C. (± 1.8° F.). All tests showing a greater variation in temperature shall be rejected.

Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. Where extreme accuracy is required, not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given below. Results obtained on samples showing evidences of foaming during the test shall be rejected.

Up to 5 per cent loss in weight, the results obtained may be considered as correct within 0.5. Above 5 per cent loss in weight the numerical limit of error increases 0.01 for every 0.5 per cent increase in loss by volatilization, as follows:

Volatilization Loss, Per Cent	Numerical Correction	True Volatilization Loss, Per Cent
5.0	±0.50	4.50– 5.50
5.5	±0.51	4.91– 6.01
6.0	±0.52	5.48– 6.52
10.0	±0.60	9.40–10.60
15.0	±0.70	14.30–15.70
25.0	±0.90	24.10–25.90
40.0	±1.20	30.80–41.20

NOTE.—If additional periods of heating are desired, it is recommended that they be made in successive increments of five hours each. When the penetration or other characteristics of the sample after heating are required, melt the residue in the container at the lowest possible temperature and thoroughly mix by stirring, taking care to avoid incorporating air bubbles in the mass. Then bring it to the standard temperature and test as prescribed in Test 9b.

In Germany, this test is performed in a container 128 mm. in diameter (128 sq. cm. surface area) and 1.5 cm. in depth (internal). Other than this, the remaining details of procedure are the same as the foregoing.[133]

An extension of this test recommended by the author, consists in heating the sample to 500° F. for five hours. This is advisable in examining relatively non-volatile asphaltic products, which would show but a fraction of a per cent loss by the foregoing method.

EVAPORATION TEST

Test 16a. A.S.T.M. Method. This test is used principally for testing road oils, in determining the so-called asphalt content, and is carried out by evaporating the specimen under carefully controlled conditions until the residue shows a penetration of exactly 100 at 77° F. (100 g., 5 sec.). The percentage by weight of residue is recorded and furnishes an indication of the quantity of constituents present which may be depended upon to contribute to the durability of the pavement. It will serve to differentiate between straight-distilled and cut-back products. This test has been standardized as follows:[134]

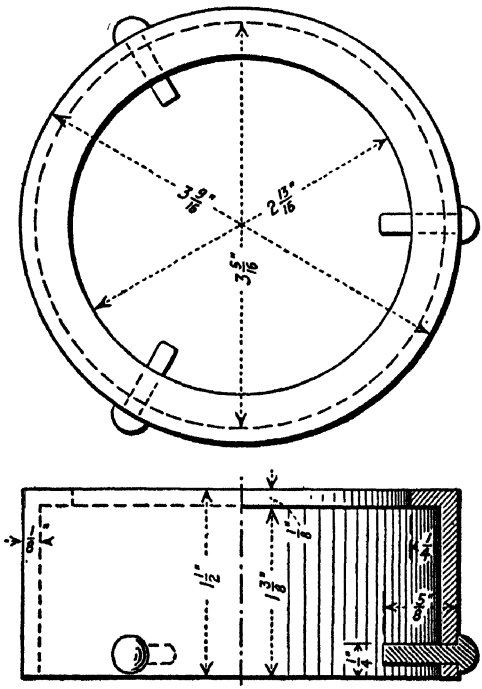
This method of test covers the determination of percentage of residue having a specified penetration at 100 g., 5 seconds, 25° C. (77° F.), obtained by heating a road oil or a semi-solid asphalt having a penetration of more than 100, at a temperature of 249 to

260° C. (480 to 500° F.). When the penetration of the residue is not otherwise stated it shall be understood to be 100. The residue obtained is available for testing as desired.

The apparatus shall consist of a container, heating bath, hot plate, and thermometer, with necessary accessory apparatus. The container in which the sample is to be tested shall be a flat-bottom, cylindrical seamless tin box, 70 mm. (2¾ in.) in diameter and 45 mm. (1¾ in.) in depth.

NOTE. The American Can Co.'s 6-oz. gill style flat-bottom, seamless ointment box, deep pattern, fulfills these requirements.

(*a*) The heating bath shall be a cast-iron air-bath permitting the immersion of the container to a depth of 1¼ in. through an



Courtesy A.S.T.M.

FIG. 264.—Cast-Iron Air-Bath.

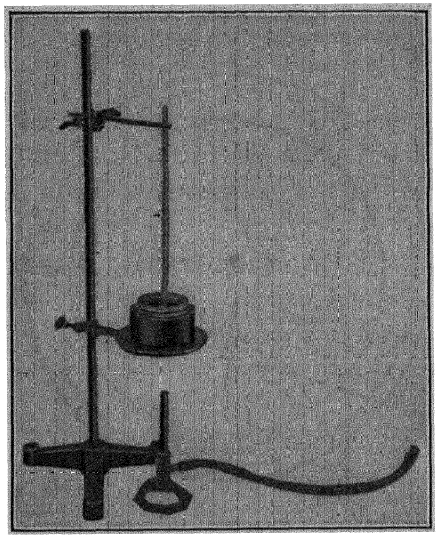
opening 1/16 in. larger in diameter than the container. It shall support the container ¼ in. above the hot plate and with at least ¼ in. free air space between the sides of the container and of the air-bath below the opening. A suitable air-bath is shown in Fig. 264.

(*b*) The air-bath shall be heated upon a suitably mounted hot plate, heated either electrically or by means of a gas flame. The

plate shall be capable of maintaining the sample continuously at the required temperature, and apparatus necessary to fulfill this requirement, such as a rheostat or gas pressure regulator, shall be provided.

The thermometer shall be a special thermometer graduated to either Centigrade or Fahrenheit degrees as specified, the ranges being — 6 to 400° C., or + 20 to + 760° F., respectively. The sample as received shall be thoroughly stirred and agitated, to insure a complete mixture before the portion for testing is removed.

One hundred grams (100.00 ± 0.10 g.) of the material to be tested shall be weighed into a tared container, which shall then be



Courtesy A.S.T.M.

FIG. 265.—Assembly of Apparatus for Evaporation Test.

placed in the air-bath in position to be heated. The thermometer shall be supported in the sample equidistant from the sides of the container and with the bottom of the bulb neither more than ¼ in. above nor touching the bottom of the container. The bulb shall be completely immersed in the sample throughout the heating. An assembly of the apparatus is shown in Fig. 265.

The sample should be heated as rapidly as possible, to prevent foaming, to a temperature of 249° C. (480° F.) and during the evaporation, the temperature shall be maintained between 249° C. (480° F.) and 260° C. (500° F.). The sample shall be stirred

with the thermometer from time to time to prevent local overheating and, to maintain a homogeneous sample, all cakes of hardened bitumen which form at the sides of the container shall be fluxed in the sample.

An experienced operator can judge approximately what percentage of residue he should obtain to secure the desired penetration. When it is supposed that the residue will show the required penetration, the bitumen on the thermometer which may be readily scraped off shall be returned to the container, which then shall be removed from the air-bath, cooled and weighed. The penetration of the residue shall then be determined in accordance with Test 9b, with the exception that the 6-oz. container specified, in which the evaporation has been conducted, shall be used instead of the 3-oz. container specified in Test 9b.

It frequently is necessary to make several trials before a residue of the required penetration is obtained. If it is determined to be greater than that required, all water shall be removed from the container and the surface of the sample, and the heating and determination of penetration may be repeated as before. Ordinarily a residue shall be considered as satisfactorily obtained when its penetration is within 15 of that desired, and its percentage by weight of the original sample shall be calculated. When it is necessary to determine more precisely the percentage of residue having the specified penetration, such a percentage shall be computed by interpolation between percentages of two residues, one having a penetration greater and one having a penetration lower than that specified. The percentage shall be reported as:

Percentage of residue of ..... penetration (determined .....) stating, first, the specified penetration, and second, the penetration actually determined for the sample tested or calculated by interpolation.

Certain types of road oil will readily form rings of hard asphalt at the side of the container. Great care should be taken that this material be completely fluxed in the sample before the penetration of the residue is determined.

Duplicate determinations should not differ by more than 1.0 per cent with the same operator nor more than 2.5 per cent between different laboratories.

DISTILLATION TEST

Test 16b. A.S.T.M. Method. The value of this test is to ascertain the adaptability of bituminous materials for a given use, generally for road treatment; also for gauging the uniformity of supply, for purposes of factory control, and most important of all, as a criterion of the quality.

According to Sharples [135] the distillation test as applied to tars becomes of value in identifying the kind used (upon determining the specific gravity of the fractions distilled), as a means of distinguishing a cut-back tar from a straight-distilled tar (upon determining the specific gravity of the fractions, their viscosity, also the fusing-point of the residue), and for detecting the presence of abnormal amounts of naphthalene.

This test has been standardized as follows: [136]

(*A*) *For Road Oils (Asphaltic- and Coal-tar Products)*: The apparatus consists of a flask, condenser tube, shield, receivers and thermometers as specified.

(*a*) Flask: The distillation flask, Fig. 266, shall be a side neck distilling flask, having the following dimensions:

Diameter of bulb, outside	86	mm. ± 1.5 mm.
Diameter of neck, inside	22	mm. ± 1.0 mm.
Diameter of tubulature, inside	10.0	mm. ± 0.5 mm.
Height of flask, outside	131	mm. ± 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature inside	93	mm. ± 1.5 mm.
Length of tubulature	220	mm. ± 5.0 mm.
Angle of tubulature	73	deg. ± 2 deg.
Thickness of tubulature wall	1.0 to 1.5	mm.

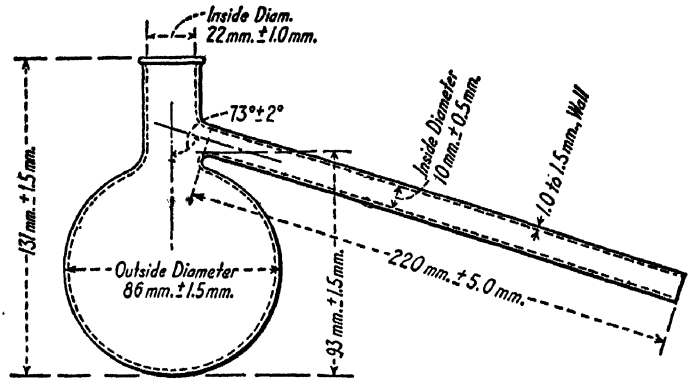
(*b*) Condenser Tube: The condenser tube shall be a suitable form of tapered glass tubing of the following dimensions:

Outside diameter of small end	12.5	mm. ± 1.5 mm.
Outside diameter of large end	28.5	mm. ± 3.0 mm.
Length	360.0	mm. ± 4.0 mm.
Length of tapered part	100.0	mm. ± 5.0 mm.

(*c*) Shield: A galvanized iron shield, lined with 1/8-in. asbestos, of the form and dimensions shown in Fig. 267 shall be used to protect the flask from air currents and to prevent radiation. The cover (top) may be of transite board made in two parts, or it may be of galvanized iron lined with 1/8-in. asbestos.

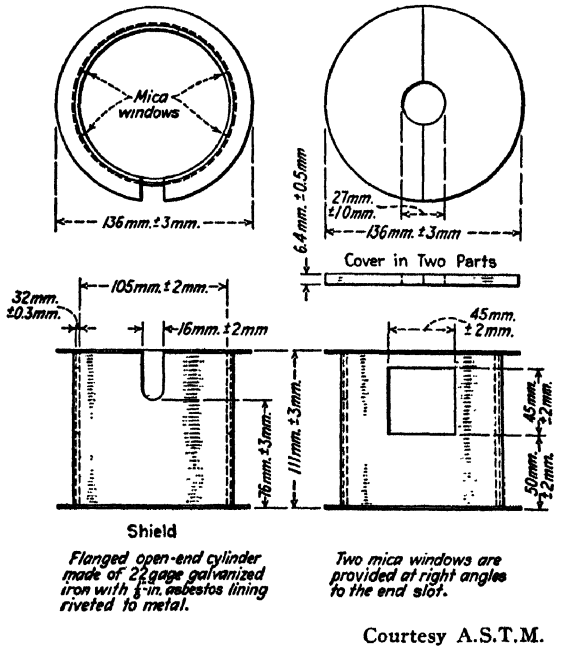
(*d*) Receiver: The distillates shall be collected in tared Erlenmeyer flasks having a capacity of 50 to 100 ml.

(*e*) Thermometer: The thermometer shall be graduated in



Courtesy A.S.T.M.

FIG. 266.—Distillation Flask.



Courtesy A.S.T.M.

FIG. 267.—Shield.

either Centigrade or Fahrenheit degrees as specified, the range being from 0 to 400° C. or 30 to 760° F., respectively.

The sample as received, shall be thoroughly stirred and agitated, warming if necessary, to insure a complete mixture before the portion for analysis is removed.

The material may be tested for distillation without dehydration, if water is present not to exceed 2.0 per cent. If water is present in excess of 2.0 per cent, the bituminous material shall be dehydrated before testing, by distilling 500 ml. in an 800-ml. copper still provided with a water-cooled condenser, the distillate being caught in a separatory funnel. When all the water has been expelled, the distillate is allowed to settle, the water drawn off and the oils returned to the residue in the still after the contents have cooled below 212° F.[137]

The flask shall be supported on a tripod or rings over two sheets of 20-mesh gauze, 150 mm. square, as shown in Fig. 268. It shall be connected to the condenser tube by a tight cork joint.

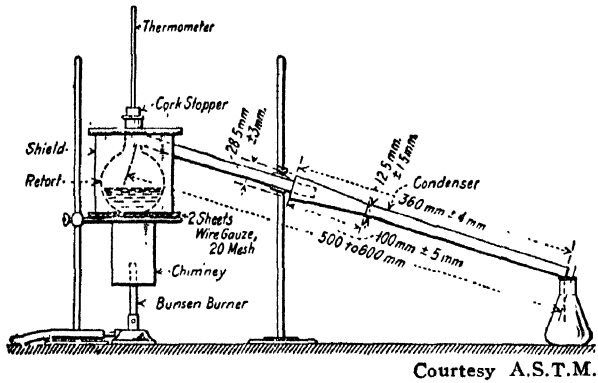


FIG. 268.—Distillation Apparatus Assembly for Road Oils.

The thermometer shall be inserted through a cork in the neck with the top of the bulb level with the lowest point of juncture of the tubulature and neck of the flask. The axis of the flask through the neck shall be vertical.

The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner should be protected from draughts by a suitable shield or chimney (see Fig. 268).

One hundred grams (100 g. ± 0.1 g.) of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from five to fifteen minutes. The distillation shall be conducted at the rate of between 50 and 70 drops per minute and the distillate collected in weighed receivers.

NOTE.—In testing creosote oils: 80 to 120 drops per minute are specified.

The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. The fractions shall be collected at the points designated by the specifications. The receivers shall be changed when the thermometer indicates the maximum temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and any oil which has condensed in the condenser tube shall be drained into the last fraction.

The residue shall remain in the flask with the cork and thermometer in position, until no vapors are visible and it shall then be weighed. If tests of the residue are required, the flask shall then be inclined so that the residue will flow around the sides, thus collecting any condensed vapors that may be on the sides of the flask, after which the residue shall be poured into a suitable receptacle and covered. If the residue becomes so cool that it cannot be poured readily from the flask, it shall be reheated to a temperature not exceeding 125° C. by holding the bulb of the flask in a suitable bath and not by the application of flame. For weighing the receivers and fractions, a balance accurate to at least 0.05 g. shall be used. During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer. The results of the distillation test shall be reported in percentages by weight of the water-free material.

(*B*) *For Cut-Back Asphaltic Products*: In testing cut-back asphaltic products, the foregoing method has been modified in the following particulars:[138]

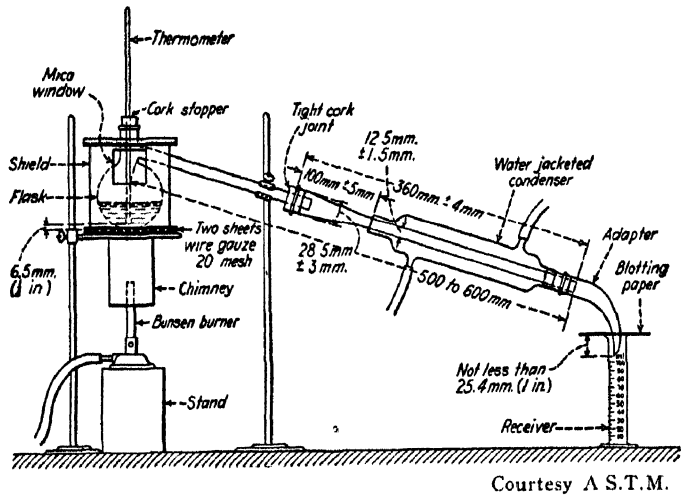
The condenser shall consist of a 250-mm. standard glass-jacketed condenser (Fig. 269), of which the following dimensions are recommended:

Length of jacket, excluding the necks	250	mm. ± 5 mm.
Outside diameter of adapter of condenser tube	23	mm. ± 1 mm.
Length of adapter	75	mm. ± 5 mm.
Outside diameter of condenser tube proper	12.5	mm. ± 0.5 mm.
Over-all length of condenser tube, including adapter	475	mm. ± 25 mm.

The adapter shall be of the curved design of heavy wall (1 mm.) and reinforced top glass, with an angle of approximately 105 deg., and with a diameter at the large end of approximately

18 mm. The outlet end shall be ground to an angle of 45 deg. ± 5 deg. with the inside vertical.

The receivers shall be graduated cylinders, of uniform diameter, with a pressed or molded base and a lipped top. The over-all height shall be not less than 24.8 cm. (9¾ in.) nor more than 26.0 cm. (10¼ in.). The cylinder shall be graduated in single milliliters to contain 100 ml., and the graduated portion shall be not less than 17.78 cm. (7 in.) nor more than 20.32 cm. (8 in.) in length. Each fifth graduation shall be distinguished by a longer line, and the graduations shall be numbered from the bottom up at



Courtesy A S.T.M.

FIG. 269.—Distillation Apparatus Assembly.

intervals of 10 ml. The graduations shall not be in error by more than 1 ml. at any point on the scale.

The flask shall be supported on a tripod or ring over two sheets of 20-mesh gauze, 150 mm. square as shown in Fig. 269. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the bottom of the bulb ¼ in. from the bottom of the flask. The axis of the flask through the neck shall be vertical. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner should be protected from drafts by a suitable shield or chimney.

The adapter shall be adjusted over the end of the condenser

tube so as to conduct the distillate into the receiver, and the top of the receiver shall be covered closely during the distillation with a piece of blotting paper or its equivalent, which shall be cut so as to fit the adapter tightly. The adapter shall extend into the receiver at least 2.54 cm. (1 in.) but not below the 100-ml. mark. Unless the laboratory air temperature is between 12.8 and 18.3 C. (55 and 65° F.) the receiver shall be immersed up to the 100-ml. mark in a transparent bath maintained between these temperatures. The condenser tube shall be clean and dry.

Two hundred milliliters (calculated from the specific gravity of the material at 15.5° C. (60° F.), of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from 5 to 15 min. The distillation shall be conducted at the rate of between 50 and 70 drops per minute. Should the sample foam the distillation rate will have to be reduced, but the normal rate shall be resumed as soon as possible. If excess foaming persists the distillation may be more easily controlled by applying the flame near the edge of the bulb instead of at the center of same. The distillate shall be collected in the specified receivers, and the volume of distillate at all specified temperatures recorded. The volume of any separated water shall also be recorded. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and the residue poured *immediately* into a 6-oz. tin box placed on its cover to prevent too rapid cooling at the bottom. Any oil which may remain in the condenser tube shall be drained into the last receiver.

As soon as no further vaporization is apparent, the residue shall be stirred to insure homogeneity, and then poured into the necessary apparatus for the required tests. During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer. Temperatures to be observed in the distillation test shall be corrected for the effect of the altitude of the laboratory in which the test is made.

If the elevation at which the distillation is to be made exceeds 1000 ft., the temperatures at which the fractions are taken shall be corrected in accordance with Table C.

TABLE C

TEMPERATURES AT WHICH CREOSOTE FRACTIONS SHOULD BE CUT TO CORRECT DISTILLATION TEMPERATURES FOR DIFFERENT ALTITUDES.

(Corrections made to the nearest 1° C.)

Elevation above Sea Level, ft.	Fractionation Temperatures for Various Altitudes, deg. Cent.					
0	200	210	235	270	315	355
1000	198	208	233	268	313	353
1500	197	207	232	267	312	352
2000	196	206	231	266	311	351
2500	196	206	230	265	310	350
3000	195	205	230	264	309	349
3500	194	204	229	263	308	348
4000	193	203	228	263	307	347
4500	193	202	227	262	306	346
5000	192	202	226	261	305	344
5500	191	201	225	260	304	343
6000	190	200	225	260	303	343

The results of the distillation test shall be reported in percentage by volume of water-free material.

(*C*) *For Coal-tar Products*: In testing coal-tar products, the foregoing method shall be modified as follows: The thermometer shall be inserted through a cork in the neck of the flask, with the bottom of its bulb 12 to 13 mm. from the surface of the oil. The exact location of the thermometer bulb shall be determined by placing a vertical rule graduated in divisions not exceeding 1 mm. back of the flask, when the latter is in position for the test, and sighting the level of the liquid and the point for the bottom of the thermometer bulb.

The following fractions are usually reported:

For Bituminous Road Oils:	For Cut-back Asphalts:	For Coal-tar Products:
Up to 170° C.	Up to 190° C.	Up to 210° C.
170 to 235° C.	Up to 225° C.	210 to 235° C.
235 to 270° C.	Up to 315° C.	235 to 270° C.
270 to 300° C.	Up to 360° C. (Note A)	270 to 315° C.
Residue	Residue	315 to 355° C.
		Residue

NOTE A.—It has been shown that a liquid temperature of 360° C. corresponds very closely to a vapor end-point of 300° C.

FLASH-POINT

The flash-point is used primarily for determining the adaptability of bituminous substances for certain definite usages, and serves as a criterion of the fire hazard. It should be at least 50° F. higher than the maximum temperature to which the bituminous substance will be subjected in the process of blending or utilization. This test is also sometimes used for gauging the uniformity of supply and for purposes of factory control. It should be noted, however, that the flash-point of a mixture is always lower than an additive result of computation.

A number of flash-point testers have been proposed,[139] of which the following are most generally used:

Test 17a. Pensky-Martens Tester. This form of tester has been standardized for determining the flash-point of fuel-oil, cut-back asphalts and other viscous materials and suspension of solids,[140] as follows:

The Pensky-Martens tester, a diagram of which appears in Fig. 270, shall include the following major parts:

(*a*) Cup. The cups shall be made of brass and shall satisfy the following dimensional specifications:

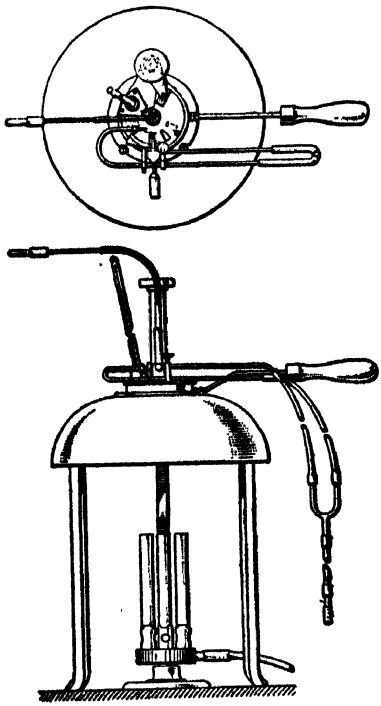
Dimensions	Inches			Centimeters		
	Minimum	Normal	Maximum	Minimum	Normal	Maximum
Inside diameter below filling mark	1.950	2.000	2.050	4.953	5.080	5.207
Difference, inside and outside diameters below filling mark	0.120	0.125	0.130	0.305	0.318	0.330
Inside height	2.150	2.200	2.250	5.461	5.588	5.715
Thickness of bottom	0.070	0.095	0.120	0.178	0.241	0.305
Distance from rim to filling mark	0.845	0.860	0.875	2.146	2.184	2.223
Distance lower surface flange to bottom of cup	1.780	1.795	1.810	4.521	4.559	4.597

The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange, but the wall thickness at the upper edge shall be not

less than 0.04 in. (0.102 cm.). The flange should be approximately 0.5 in. (1.27 cm.) wide and approximately 0.125 in. (0.318 cm.) thick. It shall be equipped with devices for locating the position of the lid on the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory.

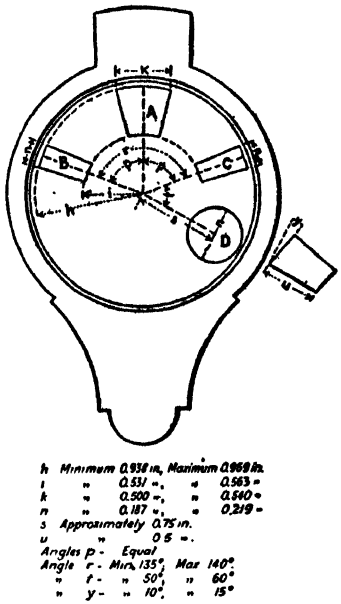
(*b*) Lid.

(*c*) Stirring Device. The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0.1 in. (0.254



Courtesy A.S.T.M.

FIG. 270.—The Pensky-Martens Tester.



Courtesy A.S.T.M.

FIG. 271.—Cover for Pensky-Martens Tester.

cm.) nor more than 0.125 in. (0.318 cm.) in diameter, mounted in the center of the cup, and carrying two two-bladed brass propellers. The blades of both propellers shall be approximately 0.313 in. (0.795 cm.) wide and shall be set at an angle of approximately 45 deg. The smaller (upper) propeller shall have an over-all diameter of approximately 0.75 in. (1.905 cm.). The larger (lower) propeller shall have an over-all diameter between 1.25 and 1.75 in. (3.175 and 4.445 cm.). The thickness of the propeller blades

shall be not less than 0.057 in. (0.145 cm.) nor more than 0.081 in. (0.206 cm.), which limits correspond respectively to No. 15 and No. 12 B. and S. gage sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0.4 in. (1.016 cm.).

The plane of the center of the upper propeller shall be 0.4 in. (1.016 cm.) below the level of the rim of the cup. The plane of the center of the lower propeller shall be 2.0 in. (5.08 cm.) below the level of the rim of the cup. The level of the rim of the cup is in effect the level of the plane part of the portion of the lower surface of the lid inside the rim.

(*d*) Cover Proper. The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim, shall be not less than 0.031 in. (0.079 cm.) nor more than 0.078 in. (0.198 cm.). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

There shall be four openings in the cover, as indicated in Fig. 271.

Opening *A* is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0.938 in. (2.383 cm.) nor more than 0.969 in. (2.461 cm.). The radius of the inner circle shall not be less than 0.531 in. (1.349 cm.) nor more than 0.563 in. (1.430 cm.). The chord of the arc of the outer circle shall not be less than 0.500 in. (1.270 cm.) nor more than 0.540 in. (1.372 cm.).

Openings *B* and *C* are equal areas, each of the same general form as opening *A* but of approximately half the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partly define opening *A*. The chord of the outer arc for opening *B* or opening *C* shall not be less than 0.187 in. (0.475 cm.) nor more than 0.219 in. (0.556 cm.). The sum of the areas of openings *B* and *C* shall not be less than 75 per cent nor more than 100 per cent of the area of opening *A*. Openings *B* and *C* shall be equally distant from opening *A* and radii drawn through each of their centers shall be at an angle of not less than 135 deg. nor more than 140 deg.

Openings *A*, *B*, and *C* need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of Fig. 271.

Opening *D* is for a thermometer collar. Its center is approximately 0.75 in (1.905 cm.) from the center of the lid and on a radius at an angle of not less than 50 deg. nor more than 60 deg. from a radius passing through the center of opening *C*. The thermometer collar shall have an inside diameter of approximately 0.5 in. (1.27 cm.). It shall be set at an angle of not less than 10 deg. nor more than 15 deg. from the perpendicular.

(*e*) Shutter. The lid shall be equipped with a brass shutter approximately 0.094 in. (0.239 cm.) thick, operating on the plane of the upper surface of the lid. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the lid between two stops, so placed that when in one extreme position the openings *A*, *B*, and *C* of the lid are completely closed and when in the other extreme position these orifices are completely opened.

(*f*) Flame Exposure Device. The flame exposure device shall have a tip with an opening 0.027 in. (0.069 cm.) to 0.031 in. (0.079 cm.) in diameter. The flame exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper, at a point on a radius passing through the center of the larger opening *A* and approximately 0.1 in. (0.254 cm.) from the outer edge of the opening.

NOTE. A pilot flame for automatic relighting of the exposure flame should be provided.

A bead 0.156 in. (0.396 cm.) in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

(*g*) Stove. Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests.

(*h*) Air Bath. The air bath shall have a cylindrical interior 1.625 in. (4.128 cm.) to 1.656 in. (4.206 cm.) deep and a diameter not less than 0.125 in. (0.317 cm.) nor more than 0.156 in. (0.396 cm.) greater than the outside diameter of the cup. The air bath may be either a flame-heated metal casting or an electric resistance element.

NOTE. If the heating element is a flame-heated metal casting it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0.25 in. (0.635 cm.) thick. The casting shall be designed so that products of combustion of the flame cannot pass up and in contact with the cup.

If the air bath is of the electric-resistance type it shall be constructed so that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

(*i*) Top Plate. The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to the bottom of the cup by not less than 0.063 in. (0.160 cm.) nor more than 0.125 in. (0.317 cm.). The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing-bushings. The spacing-bushings should be of proper thickness to define the air gap which shall be not less than 0.125 in. (0.317 cm.) nor more than 0.187 in. (0.475 cm.). The spacing-bushings shall be not more than 0.375 in. (0.952 cm.) in diameter.

(*j*) Thermometers. Two standard thermometers shall be used with the A.S.T.M. Pensky-Martens tester. The low range, "P.M. and Tag" thermometer shall be used for tests when the indicated reading falls within the limits 20 to 200° F. The "P.M. high" thermometer shall be used for tests when the indicated reading falls within the limits 230 to 700° F. For the range 200 to 230° F. either thermometer may be employed, depending on the convenience of the operator. The low-range "P.M. and Tag" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, of ranges —7 to +110° C. or +20 to +230° F., respectively. The high-range "P.M. High" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as

specified, the ranges being 90 to 370° C. or 200 to 700° F., respectively. Thermometers shall be mounted so that the bottom of the bulb is 1.75 in. (4.445 cm.) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim).

All parts of the cup and its accessories shall be thoroughly clean and dry before the test is started. Particular care shall be taken to avoid the presence of any gasoline or naphtha used to clean the apparatus after a previous test. The cup shall be filled with the oil to be tested up to the level indicated by the filling mark.

The lid shall be placed on the cup and the latter set in the stove. Care shall be taken to have the locating devices properly engaged. The thermometer shall be inserted. If it is known that the oil will flash above 220° F. the "P.M. High" thermometer may be selected; otherwise, it is preferable to start with the "P.M. and Tag" thermometer and change in case a temperature of 220 to 230° F. is reached.

The test flame shall be lighted and adjusted so that it is of the size of a bead $\frac{5}{32}$ in. (3.97 mm.) in diameter. Heat shall be supplied at such a rate that the temperature read on the thermometer increases not less than 9 nor more than 11° F. per minute. The stirrer shall be turned at a rate of from 1 to 2 revolutions per second. Application of the test flame shall be made at each temperature reading which is a multiple of 2° F. up to 220° F. For the temperature range above 220° F., application shall be made at each temperature reading which is a multiple of 5° F. The first application of the test flame shall be made at a temperature at least 30° F. below the actual flash-point. Application of the test flame shall be made by operating the device controlling the shutter and test-flame burner so that the flame is lowered in one-half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

The flash-point is taken as the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

The barometric pressure shall be observed and recorded. No corrections shall be made except in case of dispute, when the flash-point figure shall be corrected according to the following rule: For each inch (25 mm.) below 29.92 in. (760 mm.) barometric reading, add 1.6° F. to the flash-point. For each inch (25 mm.) above 29.92 in. (760 mm.) barometric reading, subtract 1.6° F. from the flash-point.

Determination of Flash-point of Cut-back Asphalts and Other Viscous Materials and Suspensions of Solids. The apparatus shall consist of the following:

(*a*) Pensky-Martens tester as described above, except that the stirrer shall be mechanically operated to stir in a downward direction at a speed of 70 to 80 r.p.m.

(*b*) Low-range thermometer as specified.

The material to be tested and the tester shall be brought to a temperature 30° F. lower than the estimated flash-point. The air space between the cup and the interior of the air bath shall be completely filled with water at the temperature of the tester and material. The temperature shall be raised throughout the duration of the test at a rate of not less than 4 nor more than 6° F. per min. With the exception of this requirement for rate of heating and the rate of stirring of 70 to 80 r.p.m., the procedure shall be the same as that prescribed in the Standard Method of Test for Flash-point by Means of the Tag Closed Tester (Test 17c).

Duplicate tests should not differ by more than 5° F.

A simplified form of Pensky-Martens tester for approximately determining the flash-point, consists of a glass beaker or metal cup having the same dimensions, namely 5.0 cm. in diameter, and 5.5 cm. in depth, filled to within 2.1 cm. of its upper rim with the material to be tested. This is supported on a sand bath and the thermometer bulb immersed in the bituminous material without, however, touching the sides or bottom. The test flame is adjusted to a 3 mm. cross-section, and the test performed exactly is described for the Pensky-Martens tester.[141]

Test 17b. Cleveland Tester. This tester has also been standardized as follows:[142]

The open-cup flash and fire test on all products except fuel oils

and those having an open cup flash below 175° F. shall be determined in the Cleveland Open Cup.

(*a*) The cup illustrated in Fig. 272 shall be made of brass and shall conform to the following requirements:

TABLE CI

	Inches			Centimeters		
	Min.	Normal	Max.	Min.	Normal	Max.
Inside diameter immediately below filling mark	2 15/32	2 1/2	2 17/32	6.27	6.35	6.43
Outside diameter below flange	2 21/32	2 11/16	2 23/32	6.75	6.83	6.91
Inside height from center of bottom to rim	1 9/32	1 5/16	1 11/32	3.25	3.33	3.41
Thickness of bottom	7/64	1/8	9/64	0.28	0.32	0.36
Distance from rim to filling mark	23/64	3/8	25/64	0.91	0.95	0.99
Distance lower surface flange to bottom of cup	1 7/32	1 1/4	1 9/32	3.10	3.18	3.26
Vertical distance upper surface flange to rim	7/64	1/8	9/64	0.28	0.32	0.36
Thickness of rim	5/64	3/32	7/64	0.20	0.24	0.28
Width of lower surface of flange	9/16	19/32	5/8	1.43	1.51	1.59

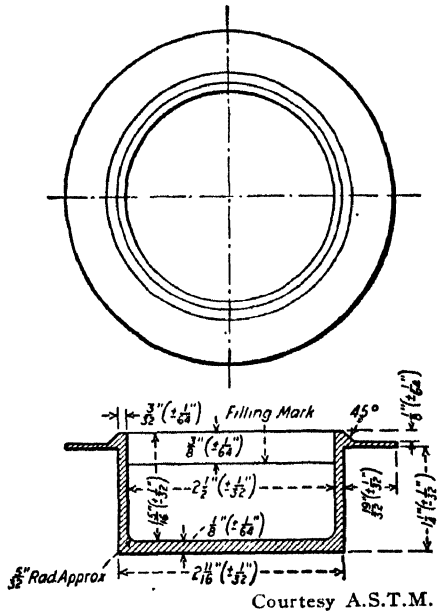
The bevelled edge of the cup shall be at an angle of approximately 45 deg. There may be a fillet of approximately 5/32 in. (0.397 cm.) radius inside the bottom of the cup.

(*b*) Heating Plate. The cup shall be supported by a metal plate (Fig. 273) 1/4 in. (0.635 cm.) in thickness and 6 in. (15.24 cm.) in width. The plate shall be of brass, cast iron, wrought iron or steel. In the center of the plate there shall be a plane depression 1/32 in. (0.079 cm.) in depth, and of just sufficient diameter to fit the cup. There shall be a circular opening 2 3/16 in. (5.50 cm.) in diameter, cut through the plate, centering with the center of the above-mentioned depression. The plate shall be covered with a sheet of hard asbestos board 1/4 in. in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the

opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the supper surface of the asbestos board.

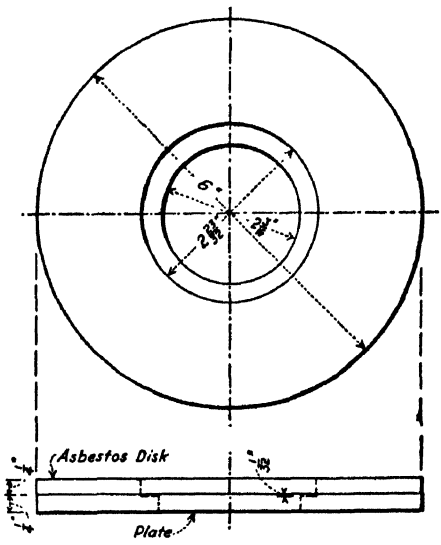
(*c*) The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being — 6 to + 400° C., or + 20 to + 760° F., respectively.

The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be 1/4 in. (0.635 cm.) from the bottom of the cup, and above a point half way between the center and back of the cup.



Courtesy A.S.T.M.

FIG. 272.—Cleveland Open Cup.



Courtesy A.S.T.M.

FIG. 273.—Heating Plate.

The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. When asphalt or other solid bituminous material is to be tested, it shall first be heated to a temperature not less than 300° F. nor more than 350° F., to render it sufficiently fluid. The cup shall then be filled with the hot fluid at this temperature in the same manner as with oil. The subsequent procedure shall then be the same for both asphalt and solid bituminous material as with oil.

The surface of the oil shall be free from bubbles. There shall

be no oil above the filling line or on the outside of the apparatus. The test flame shall be approximately 5/32 in. (0.397 cm.) in diameter.

NOTE. For purposes of comparison it is recommended that a bead of suitable light-colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately 1/16 in. (0.159 cm.) in diameter at the end and that the orifice be 1/32 in. (0.079 cm.) in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame the radius of swing shall be not less than 6 in.

The test flame shall be applied as the temperature read on the thermometer reaches each successive 5° F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 in.) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately one second.

The oil shall be heated at a rate not exceeding 30° F. per minute temperature rise, till a point is reached approximately 100° F. below the probable flash-point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50° F. before the flash-point is reached, the rate shall be not less than 9 nor more than 11° F. per minute.

The flash-point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

After determining the flash-point, the heating shall be continued at the specified rate of 9 to 11° F. per minute, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least five seconds. The method of application of the flame shall be the same as for flash-point. The temperature read at the time of the flame application which causes burning for a period of five seconds or more shall be recorded as the fire-point. The flash-point and fire-point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is

desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

Test 17c. Tag Closed Tester. This test has also been standardized [143] as follows:

The Tag Closed Tester shall be used for all volatile flammable liquids flashing below 175° F. with the exception of products classed as fuel oil, which are preferably tested with the Pensky-Martens Closed Tester. Provision is made in this method for determining the flash-point of lacquer solvents or diluents of the low flash-points.

(*a*) The Tag Closed Tester, a diagram of which is shown in Fig. 274, shall conform to the following dimensions within the limits of tolerances given:

TABLE CII

Dimension	Normal	Tolerance
Depth of water surface below top of cup, in.	1 3/32 (27.8 mm.)	± 1/64 (0.4 mm.)
Depth of oil surface below top of cup, in.	1 5/32 (29.4 mm.)	± 1/64 (0.4 mm.)
Depth of top of bulb of oil thermometer when in place below top of cup, in.	1 5/16 (33.3 mm.)	± 1/32 (0.8 mm.)
Inside diameter of oil cup at top, in.	2 1/8 (54.0 mm.)	±0.005 (0.1 mm.)
Diameter of bead on top of cover, in.	5/32 (4.0 mm.)	± 1/64 (0.4 mm.)
Weight of oil cup, g.	68	±1

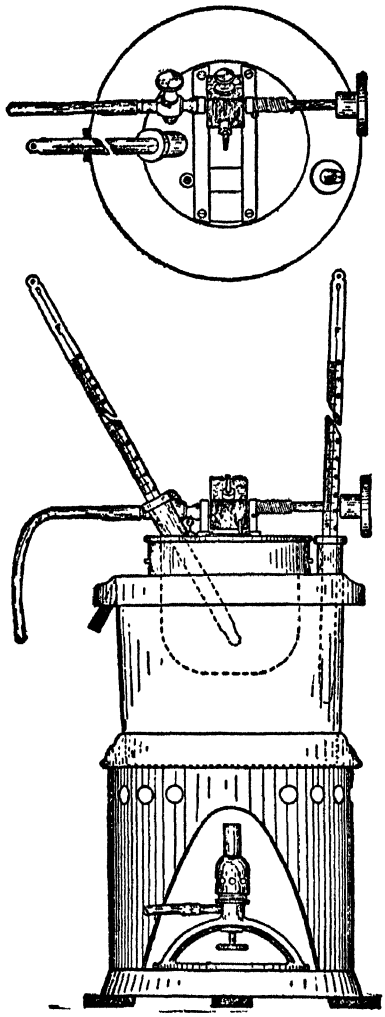
The plane of underside of cover to be between the top and bottom of the burner tip when the latter is fully depressed.

(*b*) Two thermometers are required, one for measuring the temperature of the sample under test, the other for measuring the temperature of the bath. Each of these thermometers shall conform to the following requirements, namely they shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being — 7 to + 110° C. or + 20 to 230° F., respectively.

The test shall be performed in a room or compartment darkened sufficiently to permit ready detection of the flash. Care shall be taken to have the tester level and steady. It shall be surrounded on three sides by an enclosure for protection from drafts. (A shield 18 in. square and 24 in. in height, open in front, is suggested. Tests made in a laboratory hood or near ventilators are not to be relied upon.)

Gas may be used for the test flame and for heating the water

bath. If gas is not available for the test flame, a wick of cotton cord may be inserted in the burner tip, a small quantity of cotton waste placed in the oil chamber to which the burner tip is attached and the chamber filled with signal, sperm, or lard oil. An alcohol lamp may be used for heating the water bath as a substitute for gas.



Courtesy A.S.T.M.

FIG. 274.—Tag Closed Tester. (Arranged for Use of Gas.)

The water bath thermometer shall be placed in the collar provided for it and the bath filled with water until it overflows. The temperature of the water in the bath shall be such that when testing is started it will be at least 20° F. (11° C.) below the probable flash-point of the oil to be tested.

The oil cup shall be placed in its proper position in the water bath and 50 ml. of the oil to be tested shall be measured into it, using an accurate graduate or other measuring device for the purpose. The temperature of the oil shall be at least 20° F. (11° C.) below its probable flash-point when the test is started. Air bubbles on the surface of the oil shall be destroyed, and the cover with the flash-point thermometer in place shall then be properly attached to the bath collar. The test flame shall be lighted, the flame being adjusted to the size of the small white bead on the cover.

The gas burner or alcohol lamp shall be centrally placed in the base of the tester and lighted. The flame shall be so adjusted that the temperature of the oil in the cup rises at the rate of 1.8° F. (1° C.) per min. as closely as possible, but in any case not faster than 2° F. (1.1° C.) nor slower than 1.6° F. (0.9° C.) per min.

The barometric pressure shall be recorded. If a barometer is not available, the figure may be obtained from the nearest Weather Bureau Station and an appropriate correction made for difference in altitude between such station and the laboratory. The initial temperature of the oil shall be recorded. When the temperature of the oil is 9° F. (5° C.) below its probable flash-point, the knob on the cover shall be turned in such a manner as to introduce the test flame into the vapor space of the cup, and *immediately* turned back again. The time consumed in turning the knob down and back shall be about one full second, or the time required to pronounce distinctly the words "thousand and one." The time at which the first introduction of the test flame is made and the temperature of the oil shall be recorded.

The application of the test flame shall be repeated after each 1° F. (0.5° C.) rise in temperature of the oil until a distinct flash in the interior of the cup is observed. The true flash must not be confused with the bluish halo which sometimes surrounds the test flame during applications immediately preceding the actual flash.

The time and the temperature of the oil when the flash-point is reached shall be recorded.

If the rise in temperature of the oil from the time of making the first introduction of the test flame to the time at which the flash-point is observed was more rapid than 2° F. (1.1° C.), or slower than 1.6° F. (0.9° C.) per min., the test shall be repeated, adjusting the gas burner or alcohol lamp to the proper rate of heating. It is not necessary to turn off the test flame with the small regulating valve on the cover; it may be left adjusted to the proper size of flame.

After completing the preliminary test to determine the approximate flash-point, the burner or lamp shall be removed, the oil cover lifted and the thermometer bulb carefully wiped off. The oil cup shall be removed, emptied, and carefully wiped until dry. The temperature of the bath shall be lowered by the addition of cold water until it is 15° F. (8° C.) below the flash-point of the oil as shown by the preliminary test. The oil cup shall be replaced and a fresh 50-ml. sample measured into it. The test procedure, as described above, shall then be repeated, introducing the test flame for

the first time, however, when the oil temperature is 10° F. (5.5° C.) below the flash-point obtained in the preliminary test.

Oil which has once been subjected to the flash test shall be discarded. If test is to be repeated, a fresh sample shall be used. If two or more determinations agree within 1° F. (0.5° C.), the average of these results, corrected for barometric pressure, shall be considered the flash-point. If two determinations do not check within 1° F. (0.5° C.), a third determination shall be made and, if the maximum variation of the three tests is not greater than 2° F. (1° C.), their average, after correcting for barometric pressure, shall be considered the flash-point.

Correction for barometric pressure shall be made only in cases of dispute or when the barometer reading varies more than ½ in. (13 mm.) from the standard pressure of 29.92 in. (760 mm.). When the barometer reading is below this standard pressure, add to the thermometer reading 1.6° F. (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash point. When the barometer reading is above the standard pressure, deduct 1.6° F. (0.9° C.) for each inch (25 mm.) of barometer difference to obtain the true flash-point.

Determination of Flash-point of Lacquer Solvents or Diluents of Low Flash-point. The apparatus shall consist of the following:

(*a*) Flash-point Tester, as described above.

(*b*) Thermometer. For determining the flash-points below 40° F. (4.4° C.), suitable thermometers of low range shall be used. These thermometers shall conform to the following ranges, 0° to 120° F. or — 20° C. to 50° C. These thermometers may not be used in the testing of materials flashing above 70° F. (21.1° C.).

The procedure to be followed shall be the same as that described above, except that in determining flash-points below 40° F. (4.4° C.) the bath shall be filled with brine or other low-freezing liquid instead of water.

Test 17d. Tag Open Tester. The Tagliabue tester may be used as an open tester (i.e., without the cover) for examining bituminous road oils, in which case the test is performed as described in Test 17*c*, the cover being omitted.

BURNING-POINT

Test 18. Conventional Method. The burning-point is used to supplement the flash-point, and is of value in determining the adaptability of bituminous substances for particular purposes, from the standpoint of fire hazard. The test may be performed in any of the apparatus described under flash-point (Test 17). In determining the burning-point, the cover of the tester is removed, and the heating, also exposure to the test flame continued in the same manner as for the flash-point, until the vapors ignite and continue to burn.

FIXED CARBON

This test is used solely for purposes of identification, and is especially useful in differentiating the asphaltites, the asphaltic pyrobitumens, and the non-asphaltic pyrobitumens.

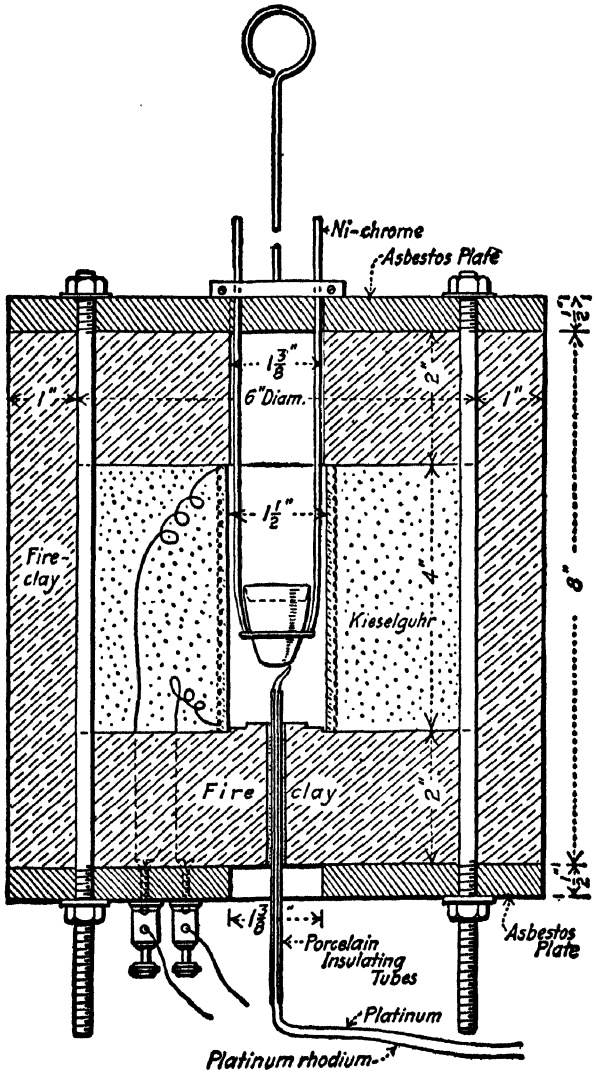
The test is a modification of the standard method for ascertaining the fixed carbon of coal.[144] There are, however, two schools of thought as to the procedure. One favors a slow initial heating of the substance to expel most of the volatile constituents without causing foaming, before applying the coal method. The other recommends the use of a crucible with a 2-mm. hole in the cover to permit the free escape of the volatile constituents and applying the full heat immediately.[145] It has also been suggested that the sample (1 gram) be covered with sand (10 grams) before ignition to inhibit entrainment of volatile matter.[146] The author favors the former procedure, patterned along the method of ascertaining the coke residue of creosote oil.

In the presence of mineral matter, the percentage of fixed carbon should be calculated on the basis of the non-mineral constituents. Mineral matter does not vitiate the results but merely acts as a diluent. Thus a pure grahamite containing 0.2 per cent mineral matter and 52.22 per cent fixed carbon, when mixed with an equal weight of clay, tested 26.33 per cent, equivalent to 52.7 per cent fixed carbon on the basis of the non-mineral constituents present.

Test 19. A.S.T.M. Method. The following method has been standardized [147] for ascertaining the fixed carbon (coke residue) in creosote or creosote coal-tar solution by coking the residue resulting

from the distillation test (Test 20) and calculating the result based on the original oil.

(*a*) *Crucible.* A platinum crucible shall be used, with tightly



Courtesy A.S.T.M.

FIG. 275.—Electric Furnace for Determining Coke Residue.

fitting cover of the inverted or capsule type having a depth of about 1 cm., provided with a hole 2 mm. in diameter at its center. The crucible shall have a capacity of 25 to 30 ml. and with cover shall weigh 25 to 30 g.

(*b*) *Furnace.* A vertical electric tube furnace of the form shown in Fig. 275 or a Bunsen or Meker burner may be used.

The residue resulting from the distillation test shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. One gram ± 0.1 g. of the residue shall be weighed into the covered crucible.

If the vertical electric tube furnace is used, the temperature of the furnace shall be controlled by a thermocouple at 950° C. ± 20° C. The crucible shall be suspended in the electric furnace for exactly 7 minutes, after which it shall be removed, cooled and weighed.

If a Bunsen or Meker burner is used, it shall have a free flame 20 cm. in height. The crucible shall set in a nichrome triangle with approximately two-thirds of its height below the triangle and with the bottom of the crucible 6 to 8 cm. above the top of the burner. Assurance of the desired temperature of 950° C. ± 20° C. may be indicated by the fusion of crystals of potassium chromate in the crucible when exposed in the specified position for the test. The crucible and contents shall be exposed to the full flame of the burner for exactly 7 minutes. The test shall be conducted in a part of the laboratory free from draughts.

The percentage of coke obtained from the residue in accordance with this test shall be calculated to percentage of total substance by the following formula:

$$\text{Coke in oil} = \frac{A \times B}{100}$$

where A = the percentage residue from oil distilled to 355° C.; B = the percentage of coke in the residue.

(D) SOLUBILITY TESTS

Test 21. Solubility in Carbon Disulfide. This test[148] is useful for purposes of identification, for ascertaining the adaptability of a bituminous substance for a given purpose, for gauging its uniformity of supply, and as a criterion of its quality (i.e., purity) and consequently its intrinsic value. Crude bituminous materials are often purchased on the basis of the percentage soluble in carbon disulfide. The presence of non-mineral matter insoluble in carbon disulfide

may be regarded as just that much inert material, and in certain cases as an indication that the material has been carelessly prepared or overheated in its process of manufacture. The mineral matter may be regarded as an adulterant. In the case of native asphalts, the larger the percentage soluble in carbon disulfide, the greater will be their intrinsic value. The percentage and composition of the mineral matter will often indicate the source of the native asphalts. Asphalts derived from petroleum are substantially free from mineral constituents, and with the possible exception of the harder grades, contain little to no non-mineral matter insoluble in carbon disulfide.

With a native asphalt containing over 10 per cent of mineral matter, it is necessary to separate the portion soluble in carbon disulfide, before ascertaining its physical characteristics, fusing-point, and sometimes fixed carbon, in which case the soluble constituents should be recovered as will be described.

Two methods will be considered, depending upon whether or not the constituents are to be subjected to a detailed analysis.

Test 21a. Where the Constituents Are Not to Be Examined Further. In this case the method recommended follows along the lines of the one standardized [149] which is substantially as follows, differing however somewhat in phraseology: The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still as described in Test 25, the water-free distillate being returned to the residue. If the material is hard and brittle, it may be ground * and dried at a temperature below the temperature of volatilization of the material, in a shallow nickel or iron dish.

A Gooch crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm. shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction shall be applied, the crucible shall be filled with asbestos † suspended in water which shall be allowed to settle partly in the

* Where it is not desirable to crush the rock or sand grains, a lump should be placed in the drying oven until it is heated through and softened at the lowest possible temperature, whereupon it may be crushed into a thin layer and dried as described.

† Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water.

crucible. A light suction shall be applied to draw off the water, leaving a firm mat of asbestos in the crucible. More suspended asbestos shall be added and the process repeated until a felt is built up that barely transmits light. The felt shall then be thoroughly washed with water, dried in a drying oven, and ignited over a Bunsen burner. The crucible shall then be cooled in a desiccator and weighed. An amount of material which shall contain approximately 1 gram of bitumen shall be weighed into a tared Erlenmeyer flask.

(*a*) *Method Used Where but Little Finely-Divided Insoluble Matter Is Present.* One hundred ml. of chemically pure carbon disulfide shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside for fifteen minutes.

The Gooch crucible shall be set up again with the suction flask and the carbon disulfide solution carefully decanted through the asbestos felt, with or without light suction as may be found necessary. No sediment shall be allowed to go onto the filter. A small amount of carbon disulfide shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The contents of the crucible shall be washed with carbon disulfide until the washings are colorless. Suction shall be applied to the crucible to remove the carbon disulfide. The crucible shall be dried in the oven at 100 to 125° C. for twenty minutes, cooled in the desiccator and weighed.

The weight of the residue in the crucible shall be recorded. (1)

Ignite the residue in the crucible at a red heat to a clean ash, cool, add a few drops of ammonium carbonate solution, re-ignite to a low red heat, cool and weigh the residue..... (2)

In case any insoluble matter adheres to the flask, it shall be dried, weighed and the increase over the original weight recorded... (3)

The filtrate containing the soluble matter shall be evaporated, the bituminous residue burned, and the weight of ash recorded. (4)

Constituents Soluble in Carbon Disulfide equal the weight of material (dry) taken for analysis, minus the sum of items (1), (3) and (4). Calculate this in per cent.

Non-Mineral Constituents Insoluble in Carbon Disulfide equal item (1), minus (2), plus item (3). Calculate this in per cent.

Mineral Constituents equal the sum of items (2) and (4). Calculate this in per cent.

(*b*) *Method Used Where a Substantial Quantity of Finely-Divided Insoluble Matter Is Present.* This procedure should be followed where the filter clogs unduly, or where the mineral matter passing through the filter exceeds 0.5 per cent. The material should be weighed out in the same way as in the foregoing, and treated with 100 ml. of chemically pure carbon disulfide.

The flask shall be loosely corked and shaken at intervals until all large particles of material have been broken down. The flask shall be left undisturbed for forty-eight hours to permit the contents to settle. The solution shall be decanted into a similar tared flask, care being taken to disturb as little of the residue as possible. The first flask shall be treated again with fresh carbon disulfide as before and left undisturbed for forty-eight hours. The solution from the second flask shall then be carefully decanted upon the Gooch crucible without use of vacuum, and this shall be followed by the solution from the first flask. The filter shall be washed with fresh carbon disulfide. The residue remaining in each flask shall be shaken again with fresh carbon disulfide and allowed to settle for twenty-four hours. The solution from both flasks shall then be decanted through the filter and the residues remaining in the flasks shall be washed again with carbon disulfide, the solution decanted and the process repeated until the washings are practically colorless. The temperature shall be maintained between 20 and 25° C.

The crucible and both flasks shall be dried at 100 to 125° C. for twenty minutes, cooled in a desiccator and weighed. The procedure from this point on is exactly the same as in the preceding. The per cent constituents soluble in carbon disulfide, non-mineral constituents insoluble in carbon disulfide, and mineral constituents shall be calculated.

The author finds that in the presence of large quantities of finely-divided insoluble matter, the method may be materially shortened by adding a weighed quantity (about twice the weight of bituminous material) of freshly ignited, long-fibered amphibole to the bituminous substance in the first flask. On shaking with carbon disulfide, the asbestos serves to dilute the insoluble matter, preventing

the latter from clogging the pores of the filter, and accordingly reducing the time of filtration.

A rapid colorimetric method depends upon the measurement of the amount of light by means of a photo-electric cell, which is transmitted through a solution of the bituminous substance, under controlled conditions, in comparison with a solution of known concentration. The solution is prepared by shaking 1 g. of the substance with a measured quantity of C.P. carbon disulfide for 20 minutes in a stoppered flask. A portion is then centrifuged to remove the insoluble constituents. The purified solution is poured into a hollow glass cell and subjected to a ray of light from a constant source. On closing the cell circuit, a microammeter in series with the photo-electric cell gives the reading of the soluble content upon a calibrated scale.* The scale is normally calibrated to give readings from 6 to 25 per cent, and the method is claimed to be accurate to within 0.1 per cent.

Test 21b. Where the Constituents Are to Be Examined Further. Extract 25 to 50 g. of the moisture-free bituminous substance with carbon disulfide as previously described, increasing the quantities of carbon disulfide proportionately. In certain cases an extractor described in Test 59 may be conveniently employed.

An alternate procedure consists in dissolving the substance in benzol or carbon disulfide and centrifuging at 3500 to 4000 r.p.m. The separated mineral matter is centrifuged a second time with clean solvent, whereupon the mineral matter is completely recovered and is entirely free from soluble organic matter.[159]

Residue Insoluble in Carbon Disulfide. This contains the non-mineral constituents insoluble in carbon disulfide (e.g., insoluble bituminous matter, free carbon and vegetable matter derived from associated soil) together with the mineral constituents retained by the filter (e.g., clay with 2 molecules of water in chemical combination,† silica, silicates, calcium and magnesium carbonates, calcium sulfate with half a molecule of water in chemical combination, iron pyrites, etc.). Dry in an oven at 220° F. to constant weight and

* The apparatus, known as the "Photo-Bitometer" is supplied by Hutchinson's Testing Apparatus, Ltd., Westminster, S.W. 1, London.

† The water of hydration in clay is held tenaciously, for none is given up during one hour's heating at 293° C., although it is expelled on heating to redness.

mix well to obtain a uniform sample. If the mineral matter is coarse, pulverize sufficient of the well-mixed material in a mortar to 200 mesh or finer for use in carrying out the examination described in sections (*a*) to (*e*) inclusive.

(*a*) To determine the water in chemical combination with the clay and calcium sulfate, heat 10 g. to bright redness in a stream of dry hydrogen or illuminating gas, in an electric combustion apparatus, catching the water in a tared calcium chloride bulb, which is reweighed at the end of the operation. The gain in weight represents the water which should be calculated in percentage.

(*b*) To determine the sulfur present in sulfides, weigh 25 g. into a small flask closed with a stopper carrying a thistle tube filled with hydrochloric acid and an outlet tube leading into a beaker of bromine water. Introduce the acid and boil until all the hydrogen sulfide is expelled into the bromine water, which serves to oxidize it into sulfuric acid. Expel the excess of bromine by boiling and precipitate with barium chloride in the usual manner. Calculate the percentage of sulfur present.

(*c*) To determine the carbon dioxide present in carbonates, weigh 1 g. into a Schroetter or Mohr alkalimeter and treat with hydrochloric acid in the usual manner. The loss in weight represents the sum of the carbon dioxide (liberated from the carbonates) and the hydrogen sulfide (liberated from the iron pyrites). Subtract the weight of hydrogen sulfide (ascertained in *b*) to arrive at the weight of carbon dioxide present, and calculate its percentage.

(*d*) To determine the non-mineral constituents insoluble in carbon disulfide,[151] weigh 5 g. into a porcelain dish, treat with an excess of concentrated hydrochloric acid, evaporate to dryness on a water bath and then dehydrate the silica by heating in an air-oven at 220° F. for two hours. Boil up with water, filter on a weighed Gooch crucible and wash thoroughly with boiling water until the filtrate no longer shows a reaction for calcium salts. Dry at 220° F. until no further loss occurs and then weigh the residue, which represents the non-mineral constituents insoluble in carbon disulfide, together with clay and its water of hydration, silica, silicates, calcium sulfate and its water of hydration, etc. Ignite to redness until all the carbonaceous matter is consumed and reweigh. The loss in weight represents the sum of the non-mineral constituents insoluble

in carbon disulfide, also the total water of hydration (originally associated with the clay and calcium sulfate). Subtract the per cent water of hydration (ascertained in *a*) to arrive at the per cent non-mineral constituents insoluble in carbon disulfide.

(*e*) To determine the iron, aluminium, calcium, magnesium, silica, sulfates, etc., weigh 2.5 g. into a platinum crucible and ignite until all the carbonaceous matter is consumed. Then fuse with potassium-sodium-carbonate, dissolve the melt in water, acidify with hydrochloric acid, evaporate to dryness to dehydrate the silica, take up with boiling water, filter on a weighed Gooch crucible to separate the silica, which is then washed with boiling water until free from salts, ignited and weighed. The filtrate is diluted to exactly 250 ml. at 77° F. whereupon 200 ml. are drawn off, oxidized by boiling with nitric acid, and the iron and aluminium precipitated with an excess of ammonia, filtered, washed, ignited and weighed together as Fe_2O_3 and Al_2O_3. Calcium, magnesium and SO_3 are then determined in the filtrate in the usual manner. The remaining 50 ml. of the original filtrate are then reduced with metallic zinc and sulfuric acid, and the iron titrated with N/10 $KMnO_4$. The Al_2O_3 is then calculated by difference. The percentage of clay present in the original material is calculated from the following formula: $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Gypsum is calculated from the following formula: $2CaSO_4 \cdot H_2O$. Potassium and sodium are determined in a separate portion.

Constituents Extracted: This contains the soluble bituminous constituents together with any chemically combined mineral constituents e.g., iron present in certain asphalts [152] and fatty-acid pitches, lead present in the sludge asphalts, copper present in certain fatty-acid pitches, etc.,* also any colloidal mineral constituents not retained by the asbestos filter (e.g., clay, silica, calcium and magnesium carbonates, etc.). Cool the solution to exactly 77° F., measure its volume and then dilute with carbon disulfide at 77° F. to the next higher 10-ml. mark. Thoroughly agitate the liquid and with a pipette transfer an aliquot portion into a tared crucible or dish. Evaporate the solvent over a steam bath, then incinerate the residue and ignite at white heat to a clean ash, until no further loss

* It is contended that in natural rock asphalts, a portion of the mineral matter is chemically united with the asphalt in the form of salts of sulfonated acids.

in weight occurs. Moisten with ammonium carbonate solution, re-ignite at a low heat (not beyond incipient redness) and reweigh. The ash represents the anhydrous clay, silica, calcium and magnesium carbonates, together with the mineral constituents originally in chemical combination with the bituminous matters. Calculate the mineral matter associated with the bituminous constituents in total carbon disulfide extract. Note that any water of hydration originally combined with the clay is not ascertained by the foregoing procedure, but the amount present is usually so small that it may be disregarded without vitiating the results. From the weight of the residue insoluble in carbon disulfide, calculate the soluble bituminous constituents by difference. Evaporate an aliquot portion of the well-mixed carbon disulfide extract to exactly the calculated weight.

It has been found that when the solvent is expelled by heating over a water-bath for one hour, the fusing-point is increased 2 to 3° C.; also that the retention of 1 to 2 per cent of very fine mineral matter does not change the characteristics of the recovered bituminous constituents to any appreciable extent.[153]

The following variations have been proposed for recovering the bituminous constituents in their unaltered state:

(*a*) *Evaporation at Atmospheric Pressure.* From the weight of the extracted mineral matter, calculate the bituminous matter by difference, and evaporate the carbon disulfide extract to exactly this weight. This may be conveniently performed by distilling and condensing most of the carbon disulfide over an incandescent light or an electric stove in a large distillation retort connected with a condenser and receiving flask. The retort shall have a thermometer inserted, with the bottom of the bulb approximately 1 in. from the bottom of the retort. The concentrated solution is transferred to a tared dish, evaporated dry on a steam bath with constant stirring, which may be conveniently accomplished with a motor-driven agitator, and the last traces of solvent expelled in an oven at 105° C. until the residue attains the calculated weight.[154]

An alternate procedure consists in distilling the filtered extract (containing a calculated amount of soluble constituents equal to approximately 100 g.) to a bulk of about 150 ml., then transferring the solution to a spherical flask of 300 ml. capacity. The evapora-

tion is completed on a water-bath which is heated from 40° C. up to the boiling-point, at which it is maintained for 5 hours, during which period the contents are stirred at intervals with a tared glass rod. Finally the flask is heated in an electrically heated oil-bath, from an initial temperature of 105° C. to a final temperature of 130° C. which is kept constant for a period ranging from 4 to 6 hours, during which the contents are kept stirred with the tared glass rod. The oil-bath is kept agitated with a motor driven paddle, which likewise causes the flask to revolve. The time of heating in the oil-bath may be determined by dissolving asphalt of known penetration in carbon disulfide and ascertaining the time required at 130° C. to bring its penetration back to its original figure.[155]

(*b*) *Evaporation under Vacuum.* The solvent is first removed by distillation as in the foregoing, until the temperature of the residue in the flask reaches 300° F. The distillation flask is then connected to a filter pump and the distillation continued under a vacuum of 180 mm. mercury, raising the temperature to 500° F. at the rate of 5 to 6° F. per minute. If the original penetration is greater than 150 at 77° F., the distillation temperature should not exceed 425° F. The residue in the flask should then be weighed.[156] This procedure will give satisfactory results, provided the bituminous substance does not contain any appreciable quantity of constituents volatile at 400° F.

The following modification is claimed to give very accurate results.[157] Dissolve the substance in C. P. carbon disulfide previously dried by contact with $CaCl_2$. The extract should contain 5 to 6 per cent of the soluble constituents, and not exceed 240 ml. in volume, which may be attained by evaporation or by dilution. A shallow, circular brass or nickel plate as illustrated in Fig. 275*a* is supported horizontally (using a spirit level) in a vacuum-desiccator. About 60 ml. of the extract are poured into the plate and distributed uniformly over its bottom, whereupon vacuum is applied to the desiccator (180 mm. mercury) and maintained for 1½ hours. This induces the evaporation of most of the carbon disulfide. Any bubbles or blisters in the residue are pricked with a knife, and the entire layer scratched with the point of the knife in criss-cross directions as illustrated, so as to break it up into ridges, thereby exposing the interior of the mass. It is then evacuated another ½ hour.

The plate is then warmed for 10 to 20 minutes at 70° C. in an oven to soften the film of bituminous substance, which is thereupon scraped out with a spatula. Then another 60 ml. of the extract are introduced in the vessel and treated in exactly the same manner. This operation is repeated four times or until all of the original extract (240 ml.) has been evaporated. In this manner, about 15

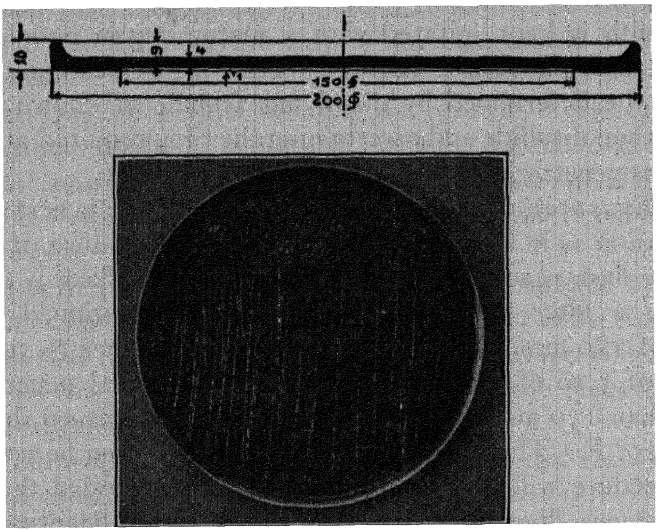


FIG. 275*a*.—Shallow Plate for Evaporation.

to 16 g. of the asphalt is collected and utilized for further tests.

This same method may be adapted to bituminous emulsions, in which case it is modified as follows: About 35 g. of the emulsion are broken by adding a few drops of conc. HCl. The separated lumps are freed from acid by kneading in several portions of water with a glass rod. The asphalt is then dissolved in about 240 ml. of carbon disulfide, allowed to stand in a separatory-funnel until most of the water rises to the surface, whereupon the solution is drawn off into a flask and shaken repeatedly with granular $CaCl_2$ at 15 minute intervals. The carbon disulfide solution is then poured off and treated as described above to recover the dissolved asphalt.

(*c*) *Evaporation in a Stream of* CO_2. In this case the carbon disulfide or benzol extract containing 50 to 75 g. of the asphalt is distilled in a wide-mouthed 250 ml. flask in a form of apparatus

shown in Fig. 276. The flask is fitted with a goose-neck vapor line of approximately 10 mm. internal diameter, connected with a condenser. The CO_2 inlet consists of a copper tube of approximately 5 mm. internal diameter, bent so that its lower end forms a circle slightly less than the diameter of the mouth of the flask, which is perforated with $\frac{1}{32}$ in. holes along its bottom to direct the gas downwardly. A thermometer is inserted through the same cork so that the lower end of its bulb reaches $\frac{1}{4}$ in. from the bottom of the flask and in the center of the circle of the copper tubing. The flask is immersed up to its neck in an oil-bath, which prevents foaming during the distillation. The rate of flow of CO_2 is measured by a differential gage or manometer connected by means of tees to

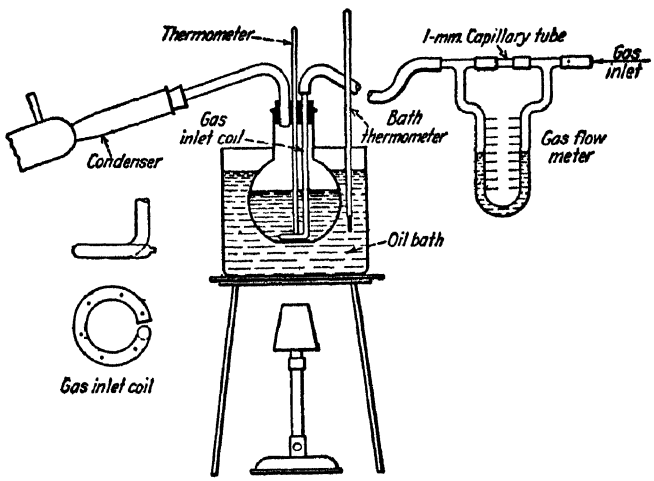


FIG. 276.—Apparatus for Evaporation in Stream of CO_2.

a 1 mm. capillary tube, which is previously calibrated. The gas is drawn from a cylinder through a pressure-regulating valve, and is bled from this through a needle-valve connected to the flow-meter. When the temperature of the asphalt reaches 300° F., the CO_2 is admitted slowly, about 150 ml. per minute, for only a few seconds at a time. This is repeated at 3 to 5° F. intervals and serves to agitate the contents of the flask and minimizes foaming. When the internal temperature reaches 325° F., the gas is admitted at the rate of 875 ml. per minute and held for exactly 15 minutes, whereupon the asphalt should be removed and poured directly into

tins or testing molds. A maximum temperature of 300° F. is recommended for asphalts softer than 100 penetration at 77° F.; likewise 350° F. for asphalts having a fusing-point of over 150° F. (R. and B.). It is important that the extracted asphalt solution be distilled on the same day that it is dissolved, otherwise considerable alteration may occur if the solution is allowed to stand for more than 10 hours, regardless of whether benzol or carbon disulfide is used, and will result in a change in the physical characteristics of the extracted asphalt.[158]

A modification of the procedure, consists in distilling off the solvent in a stream of CO_2 under reduced pressure.[159]

A rapid method for separating asphalt from asphaltic limestones and dolomites consists of the following:[160] Weigh out 10 g. of the rock asphalt and add 75 ml. of a mixture of 30 ml. concentrated HCl (sp. gr. 1.19) and 45 ml. ethyl ether. Stir 10 minutes, until the carbonates dissolve; add 75 ml. water and warm over a water-bath until the ether is expelled. Then filter and wash with warm water. Dry the filter at 105° C. and extract the filter with carbon disulfide to recover the asphalt. The extract may be treated by any of the procedures described above.

The soluble bituminous constituents, after being separated in their pure state, as previously described, may be then examined further for their physical and chemical characteristics. Due allowance should be made for the fact that any bituminous matter insoluble in carbon disulfide will be retained mechanically by the extracted mineral constituents, which with asphaltic products is usually relatively unimportant, but in the case of coal-tar products, will amount to a considerable item.

Test 22. Carbenes. The expression "carbenes" has been applied to that portion of bituminous substances soluble in carbon disulfide but insoluble in carbon tetrachloride. This term was originally proposed by Clifford Richardson.[161] This test is of value in identifying bituminous substances, gauging their uniformity of supply, for purposes of factory control, and as a criterion of their quality. Certain hard native asphalts and asphaltites, particularly grahamite, normally contain a percentage of carbenes, whereas petroleum asphalts do not show carbenes unless they are overheated

or over-blown. If more than 0.5 per cent is present in petroleum asphalts, their quality is to be regarded as questionable. Carbenes are found in tars and pitches in varying amounts.[162]

Although carbenes are found in grahamite and certain hard natural asphalts when tested as such, they disappear upon fluxing to a softer consistency. With petroleum asphalts, tars and pitches, the carbenes are of a different character, since they are insoluble in fluxes and do not disappear upon being so treated.

This test is carried out by following the same procedure as in determining the solubility in carbon disulfide (Test 21), but replacing the latter with carbon tetrachloride. The carbon tetrachloride must be free from carbon disulfide, which may be insured by distilling it under a dephlegmator, discarding any distillate below 76° C. The solvent is then filtered through calcium chloride, and any free hydrochloric acid removed by blowing dry air through it.

The procedure has been standardized as follows:[163]

An amount of material which shall contain approximately one gram of bitumen shall be weighed into a tared Erlenmeyer flask. One hundred ml. of chemically pure carbon tetrachloride shall be added to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom. The flask shall be corked and set aside in subdued light for at least twelve hours.

The Gooch crucible shall be set up again with the suction flask and the carbon tetrachloride solution carefully decanted through the asbestos felt, with or without light suction as may be found necessary. No sediment shall be allowed to go onto the filter. A small amount of carbon tetrachloride shall be used to wash down the sides of the flask and then the precipitate shall be brought onto the felt and the flask scrubbed with a feather if necessary to remove all precipitate. The contents of the crucible shall be washed with carbon tetrachloride until the washings are colorless. Suction shall be applied to the crucible to remove the carbon tetrachloride. The crucible shall be dried in the oven at 100 to 125° C. for twenty minutes, cooled in the desiccator, and weighed.

In case insoluble matter adheres to the flask, it shall be dried and weighed, and the increase in weight over the original weight shall be added to the weight of insoluble matter in the crucible.

The proportion of bitumen soluble in carbon tetrachloride shall be reported on the basis of total bitumen taken as 100:

$$\left.\begin{array}{l}\text{Proportion of Bitumen Soluble in Carbon Tetrachloride.}\end{array}\right\} = \frac{\text{Bitumen Soluble in Carbon Tetrachloride}}{\text{Total Bitumen}}$$

The difference between the percentages soluble in carbon disulfide and carbon tetrachloride respectively, represents the per cent of "carbenes."

Test 23. Solubility in Petroleum Naphtha. This test is employed mainly for purposes of identification. It is also used to a certain extent for determining the adaptability of bituminous substance for a given use, for gauging the uniformity of supply, and for purposes of factory control. As a general principle, the harder the bituminous product, the smaller will be the percentage that dissolves in petroleum naphtha. Asphaltites are relatively insoluble in this menstruum. Mineral waxes, peat-, lignite- and shale tars or pitches are largely soluble. The solubility of native and petroleum asphalts varies, depending largely upon their hardness, and also in the case of petroleum asphalts upon the extent to which the distillation has been driven. Coal-tar pitches are relatively insoluble in petroleum naphtha.

The portion soluble in petroleum naphtha has been termed "petrolenes" by some, and "malthenes" by others, whereas the nonmineral constituents insoluble in naphtha are generally referred to as "asphaltenes." [164] Asphaltenes are the characteristic constituent of asphalt, which serve to distinguish it from all other petroleum products, and are responsible for its plastic properties.[165]

It is important that the petroleum naphtha should be derived from petroleum composed entirely of open-chain hydrocarbons, and test exactly 88° Baumé, equivalent to a specific gravity of 0.638 at 60° F./60° F. At least 85 per cent by volume should distil between 95 and 150° F. The density and character of the naphtha is important, since heavy distillates, or products derived from petroleum containing unsaturated or cyclic hydrocarbons will exert a greater solvent action upon the bituminous substance.

The results will be more consistent if the petroleum spirits is first washed with fuming sulfuric acid to remove the aromatic con-

stituents. There appears to be no difference in the results if the precipitation is carried out at any temperature between 0 and 32° C. As the boiling-point of the petroleum spirits employed in making the test increases, the quantity of precipitate decreases. The fraction below 105° F. appears to give the most reliable results.[166]

Asphaltenes show increasing solubility in solvents in the order of their surface tension (e.g., ether, benzol, carbon disulfide and pyridine). Ether has been recommended as a substitute for petroleum naphtha, because it is a homogenous substance, not requiring standardization, and in addition has good flocculating properties and exerts a greater solvent action on hydroxy acids present in certain asphalts.[167]

This method is performed in the same manner as for determining the portion soluble in carbon disulfide, petroleum naphtha being substituted for the latter. Hard bituminous substances should be powdered; liquid bituminous substances flowed in a thin layer over the bottom of the flask; and semi-solid to semi-liquid substances heated until fluid and distributed in a thin layer to present a greater surface to the solvent. It is advisable not to use a stirring rod, as this causes the bituminous substance to adhere to the inner surface of the flask and to the rod itself. The operation should take place at room temperature, and away from the direct rays of the sun. The introduction of a weighed portion of long-fibered asbestos to the solution will assist in its filtration.[168]

The percentage of asphaltenes varies considerably; thus, with asphalts all having the same R. and B. fusing-point of 140° F., the following are obtained: extracted asphalt from Trinidad asphalt 37 per cent, Mexican residual asphalt 20 per cent, California residual asphalt 12 per cent, Colombian residual asphalt 16 per cent, Illinois residual asphalt 12 per cent, Texas residual asphalt 9 to 17 per cent.

Test 24. Insoluble in Benzol ("Free Carbon"). This test is generally used for testing tars and pitches for the presence of non-mineral matter insoluble in hot benzol, although sometimes carbon disulfide is used for the purpose. The test is of value for purposes of identification, for ascertaining the adaptability of the tar or pitch for a given purpose, and for gauging its uniformity of supply. Tars and pitches containing large percentages of insoluble matter, known

as "free carbon," are objectionable for certain manufacturing purposes, since the free carbon acts as so much inert matter. The term "free carbon" is a misnomer, since it is not elemental carbon, but a complex mixture of hydrocarbons of high molecular weight, containing 90.0 to 91.7 per cent carbon, 3.4 to 4.0 per cent hydrogen, 1.0 to 1.2 per cent nitrogen, 2.5 to 3.3 per cent oxygen and 0.7 to 1.4 per cent sulfur, on the ash-free basis.[169] The presence of hydrogen has been explained by the great adsorptive power of carbon in its pure state, which retains hydrocarbons tenaciously, as well as hydrogen, which is not driven off at temperatures as high as 800° C. Free carbon is more soluble in aniline or pyridine than in benzol or carbon disulfide. Selenium oxychloride exerts the greatest solvent action upon it, but unfortunately the residue cannot be freed from this solvent. It is also partially decomposed by digesting with hot fuming nitric acid.[170]

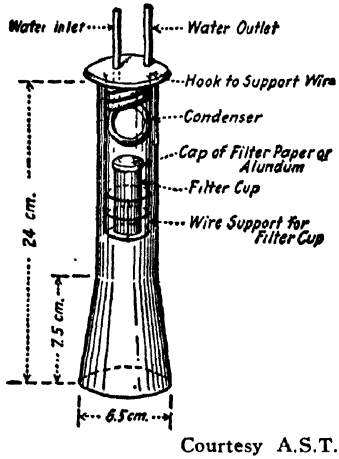
Marcusson has found that the benzol-insoluble constituents of vertical- and horizontal-retort coal-tars, amounting to 7 per cent and 24 per cent respectively, consisted of oxy-acids 8.6 per cent and 0.5 per cent; pyridine soluble resins 73 per cent and 16.3 per cent; pyridine insoluble resins 18.4 per cent and 32 per cent; and partly coked material 0 per cent and 51.2 per cent respectively.[171] The following method of extraction should be used.[172] Tars must be dehydrated before extracting, and pitches, if sufficiently hard, ground to a powder.

(*A*) *Porous Thimble Method*: The extractor shall be of a form shown in Fig. 277, or of a similar form in which the substance is subjected to direct washing by the vapors of the boiling solvent. The filtering medium shall be a flat-bottom, 30 by 80 mm. RA-98 alundum thimble. The thimble shall be suspended in the extraction flask either by a wire basket hung from two small hooks on the under surface of the metal cover of the flask, or it shall be supported by making perforations near the upper edge of the thimble and suspending from the cover by German silver or platinum wire.

An amount of material which shall contain 10.0 g. ± 0.1 g. of the substance shall be weighed into a 100-ml. beaker; 50 ml. of pure benzol shall be added and the solution stirred thoroughly. The solution shall be transferred at once to the weighed alundum thimble. The beaker shall be rinsed clean with pure benzol and the

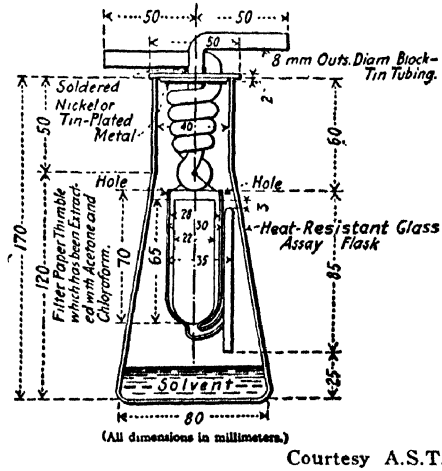
washings added to the thimble. The thimble shall then be covered with a lid of alundum ware and placed immediately in the extraction apparatus. The extractor shall contain a suitable quantity of pure benzol and shall be heated sufficiently to boil the solvent. The extraction shall be continued until the solvent descending from the thimble is colorless. The thimble shall then be dried at 105° C. ± 5° C., cooled in a desiccator, and weighed.

(*B*) *Asbestos Mat Method:* The filtering medium shall consist of a No. 3 Gooch, Coors porcelain or equivalent crucible approximately 3.5 cm. in diameter at the top, tapering to 2.2 cm. at the



Courtesy A.S.T.M.

FIG. 277.—Extraction Apparatus.



Courtesy A.S.T.M.

FIG. 278.—Extraction Apparatus.

bottom with a depth of 4.0 cm., containing a mat of acid-washed medium fiber asbestos approximately 2 mm. in thickness.

The Gooch crucible shall be placed in the suction apparatus and filled with acid-washed medium fiber asbestos suspended in water. Gentle suction shall be applied and more of the suspension, if necessary, shall be added to make a mat approximately 2 mm. in thickness. With the suction still on, the pad shall be washed with water until all small particles of asbestos are removed. The crucible shall be dried at 105° C. ± 5° C., placed in a desiccator, and weighed.

An amount of material which shall contain 10.0 g. ± 0.1 g. of the substance shall be weighed into a 125-ml. Erlenmeyer flask; 50 ml. of pure benzol shall be added and the solution stirred thor-

oughly and brought to a boil. The hot solution shall be carefully poured into the weighed prepared Gooch crucible without suction until the mat is covered. Then gentle suction shall be applied to the crucible and the remaining solution added, taking care that the mat is covered with solution at all times. The Erlenmeyer flask shall be rinsed clean with pure benzol and the washings added to the crucible. Benzol shall be added to the crucible until the descending solvent is colorless. The crucible shall then be dried at 105° C. ±5° C., cooled in a desiccator, and weighed.

(*C*) *Paper Thimble or Filter-paper Method:* A paper thimble or else two thicknesses of S. & S. No. 575 or Whatman No. 5 hardened filter-paper (15 cm. in diameter, arranged in cup shape by folding) may be used in connection with an extractor of the form shown in Fig. 278.[173] In this case the procedure is similar to that outlined in the Porous Thimble Method (*A*).

The insoluble residue of "free carbon" includes the mineral ash, which may be ascertained by incineration. Some tars (e.g. blast-furnace tars) contain substantial amounts of ash, which would otherwise introduce serious errors. It has been found that the amount of free carbon will increase with the time the solvent remains in contact with the tar or pitch. The amount of such increase varies with the particular solvent employed. In the case of carbon disulfide or chloroform, the increase is less than with benzol or toluol (or mixtures of these two) and the amount apparently reaches a maximum in about 120 hours.[174]

A method has been proposed in which 5 g. of the pitch is heated with 200 ml. tetralin in an autoclave at 240–250° C. under a pressure of 12–13 atmospheres for 2 hours. Upon cooling, the liquid is filtered through a Gooch crucible and washed successively with 50 ml. tetralin and 100 ml. benzol. The residue is dried for 2 hours at 150° C. in an atmosphere of CO_2 and weighed.[175] Anthracene oil (freed from crystallizable substances at 15° C.) has also been proposed, in which case, 5 g. of the substance are digested with 5 g. anthracene oil under a reflux condenser for 4 to 5 hours, cooled, diluted with 500 ml. benzol, filtered, washed with benzol, etc., as in the preceding.[176] Aniline has also been suggested as extracting medium, involving the digestion of 5 g. of substance with 25 ml. aniline over a steam bath for ½ hour, pouring the liquid on a porous clay

plate to absorb the solvent, washing the residue with pyridine, followed by benzol, drying and weighing.

An ingenious method has been described [177] for actually filtering the free carbon from liquid tars as follows:

A specially constructed pressure filter submerged in a hot water bath was used (Fig. 279). The filter medium, *E*, consisted of an alundum extraction shell of medium porosity (Norton RH 360–34 mm. diameter, 100 mm. high), which was cemented into the brass collar, *C*, by means of a paste of zinc oxide in water glass. The collar fitted snugly between the upper and the lower part of the outer cylinder, *O*. Lead gaskets were placed above and underneath the collar. After placing heated tar in the filter, the cap, *P*, was screwed into place and compressed air was admitted through the connection, *L*. The operating pressure was usually kept close to 50 pounds per square inch (3.5 kg. per sq. cm.). The filtrate was collected in a 500-ml. Erlenmeyer flask immersed in an ice bath. In spite of the relatively large filter area and the pressure employed, the operation of the filter was exceedingly slow. On the average, 150 to 200 ml. could be filtered before the cake had to be removed and the filter cleaned. The cycle required 6 to 8 hours.

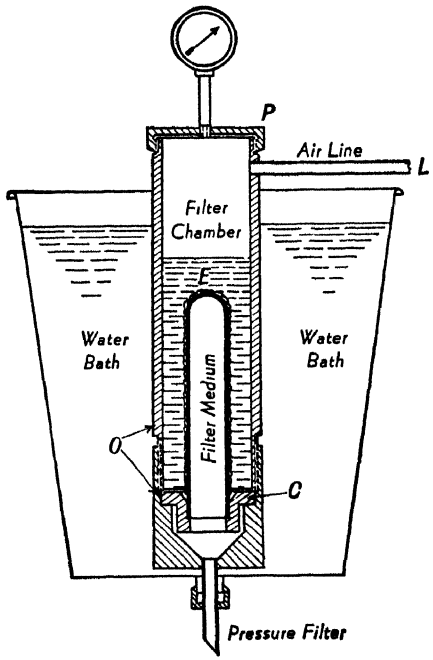


FIG. 279.—Apparatus for Removing Free-Carbon by Filtration.

After the end of the filtration, the cake was treated with two successive washings of 75 ml. each of nitrobenzene, and finally with 100 ml. of benzene. This also was done under pressure and the washings were rejected. The filter shell was then removed from the filter cylinder and placed in an air-tight container for vacuum evaporation. The evaporation was carried out over a period of at least 12 hours in a drying oven set to maintain a temperature of

175° C. The average absolute pressure, maintained by a water ejector pump, was 10 mm. After this evaporation process the filter shell was weighed and the brittle cake removed by a spatula. The filter was then suspended in an electric furnace and the remaining carbon burned. The difference in weight between the loaded and the clean filter was used as a basis for the calculation of the amount of suspended phase in the tar. However, in most cases a correction had to be made for the volatile material still present in the cake. This correction factor was determined by placing a known quantity of the detached cake in a beaker and heating on a hot plate to 250–300° C. for 2–4 hours, after which there was no further visible evolution of vapors from the cake. The amount of volatile matter

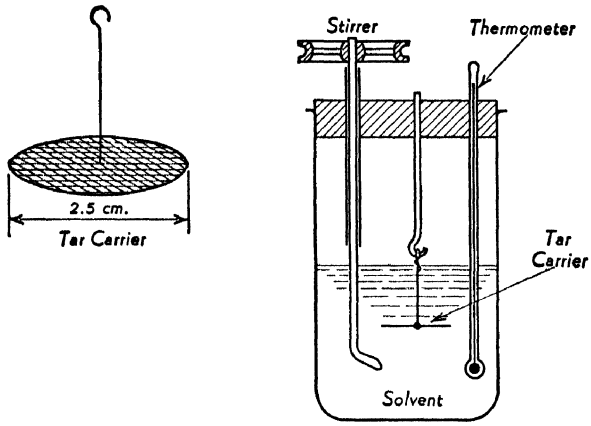


FIG. 280.—Apparatus for Solubility Determinations.

lost during this treatment was determined and used as correction factor. The amount of such vapor loss was usually less than 5 per cent of the weight of the cake.

Table CIV shows the characteristics of representative tars before and after filtering.

Test 24a. Solubility in Other Solvents. The following method has been described [177] for ascertaining the insoluble matter in tars and pitches in sundry types of solvents, including acetone.[178]

The thin film of tar is produced on a circular section of fine-mesh wire gauze (2.5 cm. in diameter) to the center of which is attached a copper wire. This copper wire serves to suspend the tar specimen in the solvent. Figure 280 illustrates the construction of this tar carrier.

TABLE CIV

Solubility of Representative Coal Tars

Type of Tar	Sample No.	Condition of Tar	Float Test at 32° C., Sec.	Sp. Gr. at 25°/25° C.	Per Cent by Wt. Dist up to 300° C.	Softening Point[a] of Dist. Residue, ° C.	No. Particles per Mg. of Tar	Quality of Suspended Phase	Suspended Phase in Per Cent by Wt. of Unfiltered Tar Determined from:				Per Cent by Wt. Insol. (Exclusive of Suspended Phase) in:				Temp. Coefficient of Viscosity[b]	Surface Tension at 90° C. by Bubble Pressure, Dynes/Cm.
									Benzene insol.	CS_2-insol.	Filtration	Microscope	CS_2	Benzene	Acetone	Ethyl ether		
Vertical-retort	1-V	Unfiltered	166 5	1 154	13 7	43 5	39,700,000	Coarse	3.15	2 99	3.6	5.5	5.44	7 43	8 97	11.09	5.94	31.5
		Filtered	160.7	1.145	13 7	40.1							5.61	7.66	9 25	11.43		31.4
	2-V	Unfiltered	162 4	1.156	14.9	45 7	53,500,000	Coarse	2.55	3.55	3.4	3.9	3 01	4 93	5 99	9 04	6 05	32 1
		Filtered	168.7	1.150	16.8	44.4							3.12	5 11	6.21	9.37		32.2
By-product coke-oven	3-C	Unfiltered	184.6	1.175	16 1	49.0	19,200,000	Very fine	0.44	1.00	1.0	0.5	3.32	4.88	7 34	12.17	6.53	35 7
		Filtered	188.0	1.172	17.1	49.0							3.36	4.93	7.42	12.30		34 9
	4-C	Unfiltered	181.9	1.195	15.5	55 2	42,800,000	Very fine	0.65	1.62	1.3	1.4	5 47	9.46	15 66	26 1	6 55	37 5
		Filtered	187 2	1.193	16 4	53 3							5 56	9.62	15.92	26 5		37 3
	5-C	Unfiltered	182.1	1 207	13 4	52.0	75,200,000	Very fine	2.76	1.91	4.0	3.8	6.12	10.66	21.1	26.5	6.49	39 8
		Filtered	171 2	1.202	17.0	52 2							6.24	10 87	21 5	27 0		39.9
	6-C	Unfiltered	173.9	1.215	10.2	39.6	114,000,000	Fine	2.88	3.37	4.2	4.0	4 07	8.99	19 02	26.2	6 50	40.1
		Filtered	167.8	1.209	11.3	40.1							4 21	9 30	19 69	27.1		40 2
	7-C	Unfiltered	183.7	1 218	13 6	51.4	91,200,000	Fine	5.17	4.71	5.7	5 6	6 47	11.41	25 2	31 2	6 35	39 8
		Filtered	186.2	1.207	17.3	56.7							6.79	11.97	26 4	32.7		39 9
Horizontal-retort	8-H	Unfiltered	184.3	1.241	14 4	54.5	107,000,000	Very coarse	17.60	17.65	23.9	24.4	5.37	7 92	11.62	18.5	6 03	38 9
		Filtered	134.6	1.196	18.9	53 7							6.52	9 62	14 12	22.5		38.6
	9-H	Unfiltered	176.3	1.270	18 9	67.6	281,000,000	Very coarse	21.67	22.32	28.9	28 2	6.63	11 93	20 8	23.1	5 26	..
		Filtered	110.1	1 202	22.9	59.4							8 53	15.36	26 8	29.7		38.2

[a] Ring and ball.

[b] $\dfrac{\text{Log } \eta_1 - \log \eta_2}{\text{Log } t_2 - \log t_1}$.

After the carrier and the crucible through which the final solution is to be filtered have been weighed, a sample of the tar under investigation is heated in a beaker approximately 3 cm. in diameter to a temperature at which the tar is moderately liquid. The carrier is then lowered into the tar until the wire gauze is covered completely by it. It is then raised out of the tar, and the excess tar adhering to the gauze is removed by rapidly twirling the wire between thumb and forefinger. If this is done properly, the coating of tar left on the gauze is so thin that it is possible to distinguish the form of the individual wires of the wire gauze.

To determine the weight of tar adhering to the gauze carrier and Gooch crucible are weighed again. From the actual weight of the tar on the carrier, the amount of solvent to be used is calculated. The dimensions of the equipment used and a series of tests showed the ratio of 0.5 gram of tar to 200 ml. of solvent to be practical.

The amount of solvent required by this ratio is finally transferred into a tall 500–ml. beaker. The carrier is attached to a hook provided on the cork stopper of the beaker, and the stopper brought into position. An air-driven stirrer, also held in position by the cork stopper, is started and the digestion continued for 45 to 60 minutes.

Figure 280 gives a view of the assembled digestion apparatus. After digestion has been completed, the carrier is placed in the Gooch crucible and the solution is filtered. If small particles adhere to the walls of the tall beaker, the filtrate is transferred back into the digestion flask, agitated, and poured again through the filter. If necessary this procedure is repeated. Further purification of the solid residue in the crucible by washing with pure solvents is not deemed advisable since they may have solvent powers different from those solvents in which some tar constituents have been dissolved. This holds true particularly in the case of solvents in which tars are soluble to only a relatively small extent.

Consequently, after completion of filtration, crucible and carrier were placed into a drying oven (110° C.) where they were kept over a period of 5 to 10 minutes. When petroleum ether was used as a solvent, the drying period could not be extended over more than 5 minutes, since the residue still contained so much volatile

material that prolonged heating caused evaporation of its more volatile portions.

Results obtained by this method are given in Table CV.

TABLE CV

RESULTS OF SOLUBILITY DETERMINATIONS

Type of Tar	Sample No.	Condition of Tar	Per Cent by Weight Insoluble in:				
			Carbon disulfide	Benzene	Acetone	Ethyl ether	Petroleum ether
Vertical-retort	1-V	Unfiltered	5.44	7.43	8.97	11.09	68.4
		Filtered	5.61	7.66	9.25	11.43	70.5
	2-V	Unfiltered	3.01	4.93	5.99	9.04	72.5
		Filtered	3.12	5.11	6.21	9.37	75.2
Coke-oven	3-C	Unfiltered	3.32	4.88	7.34	12.17	78.2
		Filtered	3.36	4.93	7.42	12.30	79.1
	4-C	Unfiltered	5.47	9.46	15.66	26.1	80.4
		Filtered	5.56	9.62	15.92	26.5	81.7
	5-C	Unfiltered	6.12	10.66	21.1	26.5	80.6
		Filtered	6.24	10.87	21.5	27.0	82.2
	6-C	Unfiltered	4.07	8.99	19.02	26.2	80.0
		Filtered	4.21	9.30	19.69	27.1	82.8
	7-C	Unfiltered	6.47	11.41	25.2	31.2	77.5
		Filtered	6.79	11.97	26.4	32.7	81.3
Horizontal-retort	8-H	Unfiltered	5.37	7.92	11.62	18.5	64.5
		Filtered	6.52	9.62	14.12	22.5	78.4
	9-H	Unfiltered	6.63	11.93	20.8	23.1	63.2
		Filtered	8.53	15.36	26.8	29.7	81.3

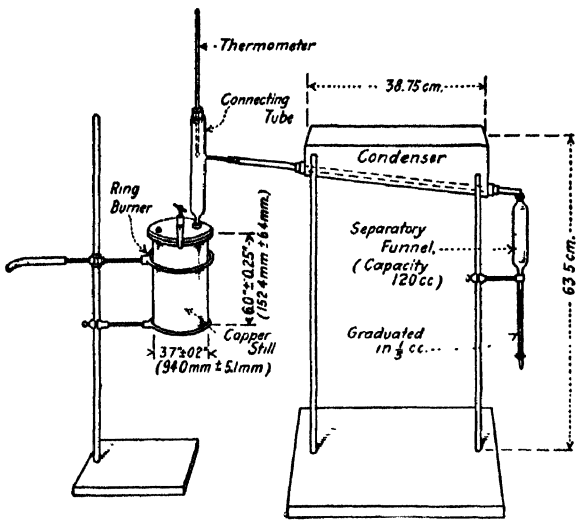
(E) CHEMICAL TESTS

WATER

The estimation of water is made in some cases for purposes of identification, and in others as a criterion of the quality. Native asphalts and tars are examined in this way to ascertain whether they exist in the crude or the dehydrated state. This test is also used for dehydrating bituminous substances to render them suitable for further examination, where the presence of water would interfere.

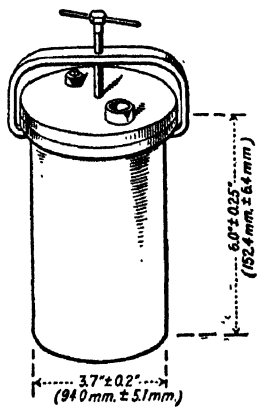
Test 25a. Substances Distilling at Low Temperatures.[179] This method is adapted to crude petroleum, tars, creosote oil and other fluid bituminous substances distilling at comparatively low temperatures.[180] The apparatus is set up as shown in Fig. 281. The copper still is provided with a removable flanged top and yoke of the form and approximate dimensions shown in Fig. 282.

The condenser consists of a copper trough carrying a straight-walled glass tube. The separatory funnel has a total capacity of 120 ml. with the outlet graduated in fifths of a milliliter. The thermometer shall be a total immersion type, graduated in either Centi-



Courtesy A.S.T.M.

FIG. 281.—Assembled Apparatus for Water Test.



Courtesy A.S.T.M.

FIG. 282.—Copper Still.

grade or Fahrenheit degrees as specified, the ranges being 0 to 400° C., or 30 to 760° F., respectively.

Measure 200 ml. of substance in graduated cylinder, and pour into copper still, allowing the cylinder to drain into the still for several minutes. Attach lid and clamp, using a paper gasket slightly wet with oil around the flange of the still. Apply heat by means of the ring burner, which shall be placed just above the level of the oil in the still at the beginning of the test, and gradually lowered when most of the water has distilled over. Continue the distillation until the vapor temperature, indicated by the thermometer with the bulb opposite the offtake of the connecting tube, reaches 205° C.

Collect distillate in separatory funnel. When the distillation is completed, and a clear separation of water and oil in the funnel has taken place, read the water by volume and draw off; and return any light oil distilled over with the water to the substance in the still. The dehydrated substance from the still shall be used for further tests.

To prevent frothing and spattering, it has been recommended [181] that the still be surrounded with a cylindrical vessel, closed at the bottom and open at the top, of a somewhat greater diameter than the still. The vessel is so adjusted that its upper rim is a little lower than the level of the bituminous substance in the retort, whereupon it is filled with water until it overflows. Heat is then applied to the shallow layer of tar above the water level, with the ring burner. The water jacket absorbs heat which would otherwise be transmitted to the lower level of the tar and cause bumping. The level of water falls gradually by evaporation, permitting the zone undergoing dehydration to fall slowly. When the water level has fallen below the bottom of the still, the contents are practically dehydrated.

A modification of the foregoing method has been suggested [182] which consists in providing the cover of the copper retort with two openings, one connected with a receptacle holding 100 ml. of tar and provided with a stop-cock to control the flow of tar into the retort. A weighed quantity of tar is introduced into the receptacle and enough run into the retort to cover the bottom, and the remainder is allowed to drip slowly into the retort. This prevents the tar frothing over.

Test 25b. Substances Distilling at High Temperatures. This method is adapted to asphalts and other bituminous substances (e.g., petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, etc.), comparatively free from volatile constituents, and incapable of distilling without suffering decomposition.

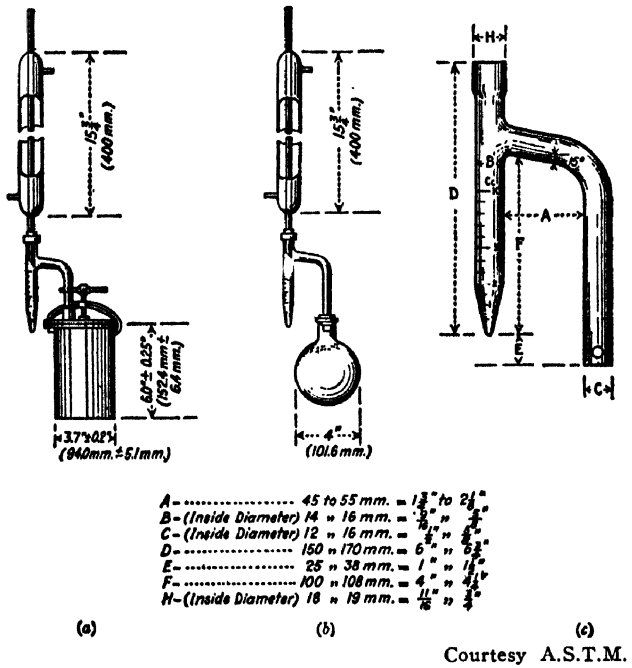
SUBSTANCES FUSING BELOW 300° F.

(*A*) *Exact Method:* When it is desired to determine the percentage of moisture without using the residue for other purposes, a convenient method consists in distilling it with a solvent.[183] The following procedure has been standardized.[184]

The apparatus shall consist of a metal still or glass flask, heated

by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method, but glass has been generally used for petroleum products and the metal still for road materials and tars.

(*a*) The metal still (Fig. 283*a*) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head



Courtesy A.S.T.M.

FIG. 283.—Apparatus for Water Test.

shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

(*b*) The glass flask (Fig. 283*b*) shall be of the short-neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

The burner used with the metal still shall be a ring gas-burner 4 in. (100 mm.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat. The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. (15¾ in.) in

length with an inner tube 9.5 to 12.7 mm. (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 deg. from the vertical axis of the condenser.

The trap shall be made of well-annealed glass constructed in accordance with Fig. 283*c* and shall be graduated as shown, from 0 to 10 ml. in 0.1 ml. divisions. The error of any indicated capacity shall not be greater than 0.05 ml. The outside diameters should be preferably 2.5 to 3.5 mm. (3/32 to ⅛ in.) greater than the inside diameters specified.

(*a*) The solvent used when testing petroleum products or bituminous materials derived from petroleum shall be gasoline, free from water, and shall conform to the following distillation requirements:

5 per cent shall distil at a temperature not below 194° F. (90° C.) nor above 212° F. (100° C.)
90 per cent shall distil below 410° F. (210° C.)

(*b*) The solvent used when testing bituminous materials derived from coal tar, water-gas tar, etc., shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements:

98 per cent shall distil between 248° F. (120° C.) and 482° F. (250° C.)

The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE. The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasized.

Procedure: When the sample to be tested contains less than 10 per cent of water, exactly 100 ml. of the material to be tested shall be placed into the still or flask and thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material. If the material is measured by volume, an accurate 100-ml. graduated cylinder shall be used and the contents transferred to the still by rinsing with one 50-ml. portion of solvent, followed by two successive 25-ml. portions of solvent, the cylinder being allowed to drain each time. When the sample to be

tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 ml. of water.

NOTE. In special cases where the water content exceeds 10 per cent and it is not desirable to reduce the size of the sample to that which will yield somewhat less than 10 ml. of water, a distilling tube receiver graduated from 0 to 25 ml. may be used. This tube shall be graduated from 0 to 2 ml. in 0.1 ml., from 2 to 5 ml. in 0.2 ml. and from 5 to 25 ml. in 0.5 ml.

The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown. The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. below the surface of the liquid in the trap after distillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from two to five drops per second. The ring burner used with the metal still should be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than an hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

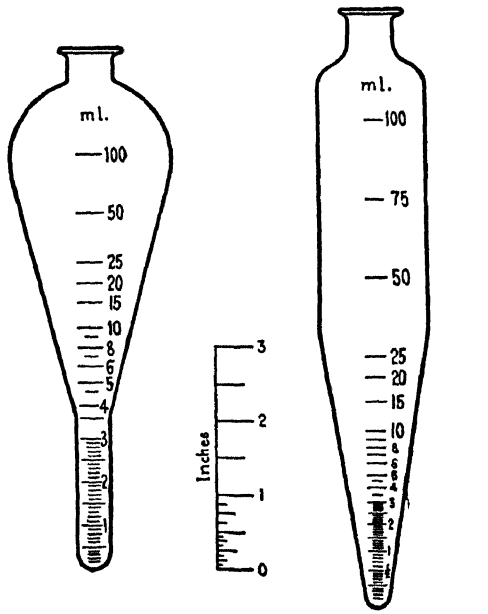
The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the volume of the sample used shall be the percentage of water and shall be reported as "..... per cent water by volume."

The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

(*B*) *Approximate Method.* The following procedure [185] gives somewhat lower readings than the actual water content, but has

the advantage of being more rapid than the foregoing test. The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself.

The apparatus shall consist of a centrifuge capable of whirling at the required speed at least two 100-ml. centrifuge tubes filled with water. It shall be of sound design and rugged construction so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned



(a) Pear-Shaped Tube. (b) Cone-Shaped Tube.

Courtesy A.S.T.M.

FIG. 284.—Centrifuge Tubes.

with water, rubber or other suitable material. The tube holders shall be surrounded during the operation by a suitable metal shield or case, strong enough to eliminate danger if any breaking occurs.

The preferred form of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 in. and a speed of at least 1500 r.p.m. or the equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper swing to give the same centrifugal force at the tips of the tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula in

which d represents diameter of swing (tip to tip of whirling tubes) of the centrifuge used:

$$\text{R.p.m.} = 1500\sqrt{\frac{16}{d}}$$

The centrifuge tubes, shown in Fig. 284, shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 ml. and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of the tube. Air-free distilled water at 20° C. (68° F.) shall be used for the calibration of tubes. The length and outside diameter are optional provided they do not conflict with the other requirements. The value of the divisions for each range with limits of error are given in Table CVI for a Pear-Shaped Tube and a Cone-Shaped Tube:

TABLE CVI

PEAR-SHAPED TUBE

Range	Scale Division	Limit of Error	Numbered
0 to 3 ml.	0.1 ml.	0.05 ml.	1, 2, 3 ml.
3 to 5 "	0.5 "	0.2 "	4, 5 "
5 to 10 "	1.0 "	0.5 "	6, 8, 10 "
10 to 25 "	5.0 "	1.0 "	15, 20, 25 "
25 to 50 "	25.0 "	1.0 "	50 "
50 to 100 "	50.0 "	1.0 "	100 "

CONE-SHAPED TUBE

Range	Scale Division	Limit of Error	Numbered
0 to 0.1 ml.	0.05 ml.	0.02 ml.	.....
0.1 to 0.3 "	0.05 "	0.03 "	.....
0.3 to 0.5 "	0.05 "	0.05 "	½ ml.
0.5 to 1.0 "	0.1 "	0.05 "	1 "
1 to 3 "	0.1 "	0.1 "	2, 3 "
3 to 5 "	0.5 "	0.2 "	4, 5 "
5 to 10 "	1.0 "	0.5 "	6, 8, 10 "
10 to 25 "	5.0 "	1.0 "	15, 20, 25 "
25 to 100 "	25.0 "	1.0 "	50, 75, 100 "

The water or oil bath shall be of sufficient depth for immersing the centrifuge tubes in a vertical position to the 100-ml. mark Means shall be provided for heating this bath to 120° F. (49° C.).

Exactly 50 ml. of 90 per cent benzol shall be measured into each two centrifuge tubes and exactly 50 ml. of the oil to be tested

shall then be added to each. The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed. The temperature of the bath shall be maintained at 120° F. (49° C.) and the centrifuge tubes shall be immersed therein to the 100-ml. mark for 10 minutes.

The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1400 to 1500 r.p.m. or the equivalent for 10 minutes. The combined volume of water and sediment at the bottom of each tube shall be read and recorded, estimating to 0.1 ml. if necessary. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 minutes, and removed for reading the volume of water and sediment as before. This operation shall be repeated until the combined volume of water and sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required. The combined total volume of water and sediment shall be read on each tube, estimating to 0.1 ml. if necessary. The sum of the two readings shall be recorded as percentage of water and sediment, centrifuge method.

With care and proper attention to details, duplicate determinations of water and sediment by this method should not differ by more than 0.2 ml. provided the centrifuge tubes are accurate and readable to this degree.

(*C*) *Alternate Method.* Where the dehydrated material is to be used for further examination, 25 g. are weighed into an Erlenmeyer flask, through which a current of dry illuminating gas is passed, and maintained at 105° C. for one hour. The vapors are led through a return condenser maintained at 50° C., and then into a weighed calcium chloride tube. When all the moisture is driven off, the calcium chloride tube is reweighed and the moisture calculated. If constituents are present volatilizing below 50° C., the return condenser should be maintained at a corresponding lower temperature.

SUBSTANCES NOT FUSING AT 300° F. In this case the material is comminuted by powdering (to about 60 mesh) or shaving, and a weighed quantity spread in a thin layer on glass and maintained in an oven at 105° C. for one hour, or until the weight becomes constant. If the substance is oxidizable in air, it should be heated in

an atmosphere of nitrogen or illuminating gas. Cool in a desiccator, reweigh and calculate the per cent moisture.

ELEMENTAL COMPOSITION

Tests 26. Carbon, and 27. Hydrogen. Carbon and hydrogen are grouped together, because both are generally determined simultaneously. These are of value in establishing the identity of bituminous materials, in connection with the corresponding percentages of sulfur, nitrogen and oxygen.

The electrical combustion method is now used almost exclusively for determining carbon and hydrogen. A weighed quantity of the material is caused to undergo combustion and the gaseous products are thoroughly oxidized by being passed over red-hot copper oxide and lead chromate. The water generated is absorbed in a weighed Marchand calcium-chloride tube, and the carbon dioxide in a weighed Liebig bulb containing a 30 per cent solution of potassium hydroxide. A furnace of the Heraeus type (Fig. 285) consisting of electrical heaters *a*, *b*, and *c*; two of which, namely, *a* and *b*, are mounted on sheave wheels running on a track so they may be moved along the combustion tube; the third heater *c* being stationary and constructed by winding an alundum or fused quartz tube 12 cm. long with No. 20 nichrome II wire, and enclosed in a cylinder packed with magnesia-asbestos. Heater *c* surrounds the lead chromate in the combustion tube. The movable heaters *a* and *b* have thin platinum foil (weighing about 9 g. in all) wound on a porcelain or fused quartz combustion tube of 30 mm. internal diameter. The large heater *b*, 350 mm. long, surrounds the copper oxide, and the smaller one *a*, 200 mm. long, heats the sample in the boat. The combustion tube *d* of Jena glass or fused silica, measuring 21 mm. external diameter and 900 mm. long, is supported by an asbestos-lined nickel trough *e*. The current through each heater is regulated by separate rheostats *f* and *g*, the heating coils *a* and *b* requiring about 4.5 amperes at 220 volts.

The furnace is arranged so either air or oxygen may be passed through the combustion tube, and is equipped with two purifying trains in duplicate (of which but one is shown in the figure) connected to the combustion tube by a Y-tube, the joint being made tight by a rubber stopper. The purifying apparatus *H* contains the follow-

ing reagents in order of the passage of the air or oxygen through them: sulfuric acid *i*, for removing any traces of ammonia; a 30 per cent potassium hydroxide solution *j*; granular soda-lime *k*; and granular calcium chloride *l*. One of the purifying trains is connected directly with an oxygen tank provided with a reducing valve for regulating the pressure, and the other being used for purifying the air supply, which is drawn through the apparatus by an aspirator connected with the other end of the combustion tube.

The first 3 cm. of the combustion tube are empty; then comes an asbestos plug (acid-washed and ignited); the next 40 cm. are filled with copper oxide gauze; then a second asbestos plug; then 10 cm. of fused lead chromate; and finally another asbestos plug 20 cm. from the end of the tube.

The absorption train consists of a 4-in U-tube *m* filled with granular calcium chloride (previously saturated with carbon dioxide) to absorb the moisture. This in turn is connected to a Vanier potash bulb *n* containing a 30 per cent potassium hydroxide solution and granular calcium chloride. The potash bulb is connected with an aspirator through the guard-tube *o* containing granular calcium chloride and soda-lime. A Mariotte flask *p* serves to keep the suction constant.

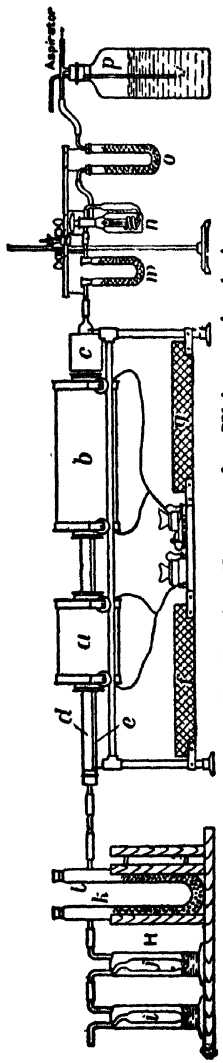


FIG. 285.—Combustion Furnace for Ultimate Analysis.

It is important to see that all connections are made tight. Before starting a determination or after any changes in chemicals or connections, a blank test should be run by aspirating 1 liter of air through the apparatus, which is heated in the same manner as though a determination were being made. If the Vanier bulb *n* or the calcium chloride tube *m* show a change in weight of less than 0.5 mg. each, the apparatus may be considered in a satisfactory condition.

Approximately 0.25 g. of the bituminous substance is carefully weighed into a porcelain or platinum boat and transferred to the

combustion tube which should be cool for the first 30 cm., the copper oxide at a bright-red heat, and the lead chromate at a dull-red heat. The boat should be introduced rapidly near the asbestos plug at the beginning of the copper oxide, the stopper connecting with the purifying train replaced and pure oxygen passed through at the rate of 3 bubbles per second. The current is *gradually* turned on heating coil *a*, which at the start should be at the right of the boat. By manipulating the rheostat, and gradually pushing the coil towards the boat, the evolution of volatile matter is carefully controlled to prevent too rapid an evolution of gas and tar, which may either escape complete combustion or be driven back into the purifying train. The heat should accordingly be increased slowly by manipulating the rheostat, until the sample ignites, whereupon the temperature may be increased rapidly. Any moisture collecting in the end of the combustion tube or in the rubber connection joining it to the calcium-chloride tube *m* is driven into the latter by carefully warming with a hot tile. After the sample ceases to glow, the oxygen is continued for 2 minutes, whereupon the heat is turned off, and 1200 ml. air aspirated through the train. The absorption bulbs are disconnected, wiped clean, allowed to cool and weighed. The percentage of carbon is equal to the increase in weight of the KOH bulb (*n*) multiplied by 27.27 and divided by the weight of the sample. The percentage of hydrogen is equal to the increase in weight of the $CaCl_2$ tube (*m*) multiplied by 11.19 and divided by the weight of the sample.[186]

Test 28. Sulfur. This test is also used for differentiating between and identifying bituminous substances.

(*a*) *Quantitative Test:* A number of methods have been proposed for this purpose, but the most rapid and accurate one consists [187] in igniting the substance in an oxygen bomb as follows:

The oxygen bomb shall have a capacity of not less than 300 ml., and shall be of a design or construction such that no leaks shall occur at any pressure or temperature generated during the test and such that when the bomb is open liquid contents can be easily and completely drained. The inner surfaces shall be of materials that are chemically and physically resistant to the process or products of combustion. The gaskets, insulating materials, etc., shall be, as far

as possible, physically and chemically resistant and in no event shall they undergo any reaction which would increase or decrease the sulfur content of the bomb liquors.

The oil cup shall be of platinum, glazed silica, or other suitable material, with a capacity of not less than 2.5 ml. nor more than 5.0 ml. If a platinum oil cup is used, the fuse wire shall be of platinum; if a glazed silica oil cup is used, the fuse wire may be of either platinum or iron. No. 35 B. & S. gage is a convenient size.

(*a*) The distilled water and all reagents should be sulfur free, but in such cases where it is necessary to employ reagents not sulfur free, blanks shall be run and the figures thus obtained used to correct the results of actual determinations.

(*b*) Barium Chloride. The barium chloride solution shall contain 100 g. of $BaCl_2 \cdot 2H_2O$ per liter.

Twenty milliliters of distilled water shall be placed in the bottom of the bomb. From 0.6 to 0.8 g. of the oil to be tested shall be placed in the weighed oil cup and the weight of this charge shall be determined to an accuracy of at least ±0.002 g. The cup shall be placed in the proper position in the bomb, the ignition mechanism arranged and the bomb closed. Oxygen shall be admitted slowly until a pressure is reached as indicated by the following table:

Capacity of Bomb, ml.	Minimum Gage Pressure Atmospheres
300 to 350	40
350 to 400	35
400 to 450	30
450 to 500	27.5
Above 500	25

The leads from the firing circuit shall be attached, the bomb placed in a bucket of cold water, and ignited. The bomb shall be allowed to stand in the water for 10 minutes and shall then be removed. The valve of the bomb shall be opened, allowing the gas to escape at an approximately even rate so that the pressure is reduced to atmospheric in not less than 1 minute. The bomb shall be opened, and all parts of its interior, including the oil cup, rinsed with a fine jet of distilled water. All washings, which should not amount to more than 350 ml. shall be collected in a beaker. Particular care should be taken not to lose, by splashing, or otherwise, any of the

liquid contents of the bomb. The washings shall be filtered through a washed "qualitative" filter paper. The filter shall be washed thoroughly. Two milliliters of concentrated HCl and 10 ml. of saturated bromine water shall be added to the filtrate. The solution shall be evaporated to about 75 ml. on a steam bath or hot plate. Ten milliliters of hot barium chloride solution shall be added in a fine stream or drop-wise to the hot solution, stirring during the addition and for two minutes afterward. The solution shall be allowed to stand overnight, or shall be kept hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for another hour, while cooling. The supernatant liquid shall be filtered through an "ashless quantitative" filter paper, the precipitate washed with water, first by decantation, then on the filter, till free from chloride. The paper and precipitate shall be transferred to a suitable weighed crucible, dried at low heat till moisture is evaporated, the paper charred (without flaming), and finally ignited at a good red heat till the precipitate is just burned white. A satisfactory means of accomplishing these operations is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition will usually occur at the desired rate. After ignition is complete, the crucible shall be allowed to cool to room temperature, and weighed. The use of a desiccator is not recommended.

From the increase in weight of the crucible the percentage of sulfur shall be calculated from the formula:

$$\text{Percentage of sulfur} = \frac{\text{grams of } BaSO_4 \times 13.734}{\text{grams of oil used.}}$$

The percentage of sulfur obtained by the same operator with the same apparatus should not differ by more than $0.02 + 0.04A$; while the percentages obtained by different operators in different laboratories should not differ by more than $0.02 + 0.08A$ (A represents the average of the percentage obtained).

A modified procedure consists in igniting 1 g. of the substance with 1 g. of an "accelerator" (potassium chlorate) and 0.2 g. of benzoic acid in a bomb calorimeter. The bituminous substance may be conveniently weighed on a small lump of chemically pure cotton (free from sulfur) and placed on a small platinum cone, which in

turn is suspended from a copper wire. The cotton is connected with a thin platinum wire forming a short-circuit between the suspended copper wire and the return conductor.[188]

Another quantitative method, which is recommended as an alternative, where the bomb-calorimeter is not available, consists in weighing 1 g. of the substance in a 100 ml. porcelain or quartz crucible, adding 5 ml. concentrated nitric acid saturated with bromine, covering with a watch-glass, digesting one-half hour on a steam bath, cooling, adding 1–2 g. anhydrous sodium carbonate small portions at a time, thoroughly mixing the contents, heating in an air-oven at 100° C., and then igniting over a low flame, gradually increasing the heat until all the organic matter has been consumed. Cool, place in a beaker, add 200 ml. water, digest on a steam bath until dissolved, filter, wash, acidify with hydrochloric acid, using congo red as indicator, adding 2 ml. acid in excess. Warm, add 10 ml. barium-chloride solution (100 g. per liter), digest on a steam bath and continue as described above.

(*b*) *Qualitative Test.* A rapid test for detecting the presence of sulfur in cyclic bodies containing sulphur in ring formation (e.g., in asphalts, asphaltites, coal-tar pitch, etc.) consists [189] in separating the saponifiable constituents (which interfere with the reaction) from 10 g. of the substance by Test 39, or by dissolving in 25 ml. benzol with gentle heating, cooling and adding 30 ml. N/2 alcoholic potash, shaking, and then rapidly diluting with 200 ml. 96 per cent alcohol. After standing a short time, the liquid (which should test alkaline to phenolphthalein) is decanted. The residue is washed with alcohol, dried on a water bath and finally at 105° C. It is then heated with 100 ml. of ether under a reflux condenser, and a few lumps of granular calcium chloride are added. After cooling, the liquid is filtered into a test tube to remove any insoluble matter present, and the solution mixed with 20 ml. 2 per cent mercuric bromide ($HgBr_2$) in ether, and allowed to stand overnight. If a precipitate forms, it is filtered off, washed with ether, and dissolved from the filter paper with warm benzol. If any sulfur-bearing bituminous substances are present, including petroleum or native asphalts, the precipitate will dissolve in the benzol forming a dark brown solution (any mercurous bromide presents remaining undissolved). On evaporating the benzol, the mer-

curic-bromide-sulfur compound is deposited as a dark brown to black brittle mass.

A confirmatory test consists in heating the mercuric-bromide-sulphur compound with a few cubic centimeters fuming nitric acid on a water bath until it evaporates to dryness. The residue is dissolved in water and tested qualitatively for sulfur (by precipitating with barium chloride), for bromine (by precipitating with silver nitrate), and for mercury (by precipitating with ammonia and ammonium sulfide) respectively. Any sulfur compounds present in fatty-acid pitch (e.g., derived by heating with sulfur, or the like), unlike those present in natural or petroleum asphalts, are not precipitated by mercuric bromide, and this reaction may accordingly be used to advantage for detecting the presence of asphalt in fatty-acid pitches. It is claimed that 10 per cent of asphalt in fatty-acid pitch may be detected in this manner.

Test 29. Nitrogen. This determination is also used for identifying bituminous products, and the procedure ordinarily employed constitutes a modification of the well-known Kjeldahl-Gunning method.[190]

One to five g. of the bituminous material, which should be finely powdered when sufficiently hard, is boiled with 150 ml. of concentrated sulfuric acid, 50 g. of potassium sulfate, 2.5 g. mercuric oxide and 2.5 g. copper sulfate in a 500-cc. Kjeldahl flask for thirty minutes to two hours, depending upon how long it takes until the liquid attains a clear green color. The boiling should be continued at least two hours after the solution reaches the straw-colored stage, the total time required ranging from three to four hours. After the solution has cooled, a few crystals of potassium permanganate are added to insure complete oxidation. When thoroughly cool, the solution is diluted to 200 ml. with cold water, again cooled, and the following solutions added: 25 ml. of a 4 per cent solution of potassium sulfide to precipitate the mercury; 1–2 g. of granular zinc to prevent bumping; and finally enough saturated sodium hydroxide (usually 80–100 ml.) to make the solution distinctly alkaline, using phenolphthalein as indicator. The danger of losing ammonia may be minimized by holding the flask in an inclined position while the sodium hydroxide solution is being added and carefully running the alkaline solution down the side of the flask so it will form a layer

below the acid solution. The flask should then be at once connected with the condensing apparatus, and the solution mixed by gently rotating the flask.

The ammonia is then distilled into 10 ml. of standard sulfuric acid solution at the rate of 100 ml. per hour, until 150–200 ml. of distillate have passed over. The distillate is then titrated with standard ammonia or caustic soda solution, using cochineal as indicator with the former, or methyl orange with the latter (20 ml. standard ammonia of caustic soda solution are equal to 10 ml. of standard sulfuric acid, and also equivalent to 0.05 g. nitrogen).

A blank determination shall be made in exactly the same manner as described above, except that 1 g. of pure sucrose (cane sugar) shall be substituted in place of the sample. The nitrogen found in this blank determination shall be deducted from the result obtained with the sample.

Test 30. Oxygen (in Non-mineral Matter). There being no satisfactory direct method for determining oxygen, it is computed by subtracting the sum of the percentages of hydrogen, carbon, nitrogen, sulfur, water and ash from 100 per cent. The result so obtained is affected by all the errors incurred in the other determinations, and especially by the change in weight of the ash-forming constituents on ignition. Iron pyrites will absorb oxygen from the air and change to ferric oxide, increasing the weight of ash, and thereby causing a negative error in the oxygen, equivalent to three-eighths of the pyritic sulfur. Any calcium carbonate present will tend to absorb sulfur combined with the bituminous constituents. On the other hand, there is always a loss on ignition of "water of composition" from the clayey and shaley constituents, also carbon dioxide from carbonates, etc., which tend to compensate for the absorption of oxygen.[191]

A more correct value is obtained by making the corrections indicated in the following formula:

$$\text{Corrected Oxygen} = 100-[(C-C')+(H-H')+N+H_2O+S'+\text{Corrected Ash}]$$

Where C equals the total carbon; C′ the carbon of carbonates; H the total hydrogen less hydrogen of water; H′ hydrogen from water of composition in clay, shale, etc.; N the nitrogen; H_2O the

moisture as found at 105° C.; S′ the sulfur not present as pyrite or sulfate; "Corrected Ash," the mineral constituents originally present, which for most purposes may be calculated with sufficient accuracy by adding to the ash as found, five-eighths of the weight of pyritic sulfur, the CO_2 of carbonates, and the water of composition of clay, shale etc.

MOLECULAR WEIGHT

Test 30a. Freezing-Point Method. This is determined by the usual Beckmann freezing-point method in a benzol or nitrobenzol solution, as described in any of the standard text books on physical chemistry. Sharp temperature readings can be obtained to 0.001°, and the weight of solvent should be recorded to within 0.01 g. This cryoscopic method is claimed to be accurate to ± 1.0 to 1.5 per cent.[192]

The following molecular weights have been reported:

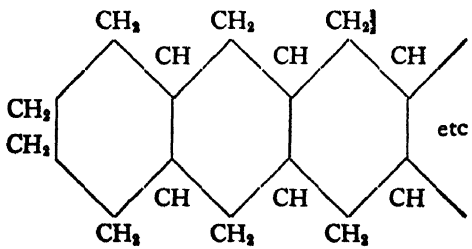
Trinidad asphalt (soluble portion)	1131.8
Bermudez asphalt (soluble portion)	620.4
Gilsonite	4251.5

The molecular weight (M) may also be ascertained from the viscosity (v) at 120° C. (at which temperature the melted substance behaves as a true solution), by means of the following formula:

$$\log v = C.k.M$$

where C = the concentration, and k = a constant. Tests indicate that asphalts have a molecular weight in the neighborhood of 1800 at 120° C.[193]

Since asphalts have a polycyclic structure of the following typical formation, it may readily be calculated that Trinidad asphalt will consist of between 12 and 16 rings:



TAR ACIDS

This test is used to ascertain the quantity of phenolic, cresylic, and other acid derivatives present in tars and pitches, especially those derived from non-asphaltic pyrobitumens, also from wood and bone. It is of value in establishing the quality for certain purposes, also as a means of identification. Two methods are employed for the purpose: [194]

Test 31a. Contraction Method. One hundred ml. of tar, or 100 g. of pitch are weighed into a tared Engler flask, and distilled by the flask method (Test 16b). With tars the distillation is continued until 95 per cent has been distilled off, and in the case of pitches it is stopped when the vapor temperature reaches 400° C. The time of distillation should occupy about twenty minutes, and the condenser tube heated to prevent the distillate from solidifying in it. The distillate is caught in a separatory funnel, the lower portion of which is graduated. This is immersed in water at 60° C. until no change in volume takes place, and a reading taken, whereupon 50 ml. of a 10 per cent caustic soda solution are added, shaken, allowed to settle, and the clear soda drawn off. The contents are brought again to 60° C., and the loss in volume noted. Shake with another 30 ml. of soda, and observe whether there is any further diminution in volume. If so, repeat until no further shrinkage occurs. The total shrinkage represents the *tar acids* present in the distillate.

NOTE. The results obtained by this method are slightly higher than the true tar-acid content, for the reason that the soda withdraws certain compounds which are not subsequently liberated from the soda solution in the form of an oil, which substances are evidently acid in nature but not phenolic bodies.

Test 31b. Liberation Method. The material shall be distilled as prescribed in Test 31a. The distillate shall be shaken in a separatory funnel with successive 50-ml. portions of 10 per cent caustic soda until no more tar acids are removed. The well-settled separated carbolate shall be acidified in a small beaker with 40 per cent sulfuric acid, taking care to keep the mixture cool at all times. If the content of tar acids is under 5 per cent, use the water in the separatory funnel and measure into it 10 ml. of solvent naphtha (xylol). The liberated tar acids and sulfate solution are then poured through this layer of naphtha several times, drawing the

material off at the bottom of the funnel into the original beaker and pouring it back into the top of the funnel. This washes out the beaker and allows all the tar acids to be absorbed by the naphtha. The funnel shall then be allowed to stand until the layer separates perfectly clear, when the sulfate solution shall be drawn off and the increase in volume of the naphtha taken as the dry tar acids present. When the content of acids is over 5 per cent, the same procedure can be used, measuring 65 ml. of naphtha into the separatory funnel. This method gives approximately the same amount of tar acids that can be recovered in practice, and averages about 90 per cent of the results obtained by the preceding method.

NAPHTHALENE

Test 32. Conventional Method. Naphthalene is present in tars and pitches generated at high temperatures, including those derived from coal. It is produced by the condensation of two or more hydro-carbon molecules accompanied by the elimination of hydrogen. Tars used for impregnating felt should not contain more than 3 per cent naphthalene, as it will otherwise crystallize out on the surface of the sheet and gradually evaporate. Naphthalene is ascertained in the following manner. The oil which is unacted upon after extraction of the tar-acids as described in Test 31, is placed in a copper beaker and cooled with stirring to 40° F. and held at that temperature for fifteen minutes. The separated naphthalene shall then be quickly filtered on a paper in a perforated funnel, using suction, and the oil removed from the solids as quickly as possible. The solid cake shall then be removed from the filter with a spatula and pressed *repeatedly* in a letter-press between strips of filter paper until only a trace of oil is given up to the paper. The solids shall then be weighed and the percentage calculated.

SOLID PARAFFINS

Until recently, it was considered that the presence of paraffin was an indication of the quality of asphaltic products, and many specifications stipulated the maximum percentage permissible. It is now generally conceded, however, that there is no rational relation between the solid paraffins in asphaltic products and their quality. The determination of paraffin is therefore of value only for

purposes of identification. Traces of solid paraffins are found in asphaltites, natural asphalts, and in asphalts produced from strictly asphalt-base petroleums. On the other hand, more or less paraffin is present in asphalts derived from non-asphaltic and semi-asphaltic petroleums. It is absent in tars and pitches derived from high-temperature distillation processes.

Test 33. Holde's Method. The following method does not give absolute figures, since it merely discloses the paraffin hydrocarbons which are *solid* at room temperature, without taking the liquid paraffins into consideration; nevertheless the results are of value for purposes of comparison.[195] Weigh 50 g. of the material in a tared 6-oz. glass retort, and slowly distil until nothing but a residue of coke remains. The distillation should take in the neighborhood of forty-five minutes from the time the first drop comes over. The distillation is conducted by heating at the rate of 40 to 50° F. per minute to 450° F., followed by a rise of 15° F. per minute to 500° F.[196]

The distillate is caught in an Erlenmeyer flask, and weighed. Either 5 or 10 g. of the well-mixed distillate, depending upon the quantity of solid paraffins present in the crude material, are transferred into a large test tube and dissolved in 25 ml. of absolute ethyl ether and 25 ml. of absolute ethyl alcohol. A similar mixture containing 25 ml. each of ether and alcohol is made up, and this together with the oil solution is cooled separately to exactly 0° F. for one-half hour in a mixture of ice and salt (to which if necessary calcium chloride may be added). The oil solution is then rapidly filtered through a weighed Gooch crucible similarly maintained at 0° F. by a jacket of ice and salt, and washed with 50 ml. of the cooled ether-alcohol mixture. A simple and convenient apparatus consists of an inverted bottle 6 in. in diameter, having the bottom cut off, and attached to the same rubber stopper which supports the funnel holding the Gooch crucible. The space between the bottle, the crucible, and the supporting funnel is packed with the ice and salt mixture. The Gooch crucible is then removed, the outside wiped clean, placed on a tared glass and dried in an oven at 80° C. until the last traces of ether and alcohol are evaporated. The residue is weighed, and the percentage of solid paraffins calculated in the original 50 g. of substance taken for analysis.

Various substitutes have been suggested for the ether-alcohol mixture, including methyl-ethyl-ketone (butanone) containing 1.3 per cent water, having a specific gravity of 0.812 at 20° C.;[197] also nitrobenzene used at 32° C.[198]

It is a mooted question whether the solid paraffins ascertained by the foregoing method are originally present as such in natural asphalts and asphaltites, or are generated during the distillation process through the destructive decomposition of other non-paraffinic components.[199]

An alternate method used with success by the author, consists in dissolving 10 g. of the substance in the smallest amount of benzol, applying heat if necessary, adding 200 ml. of warm 88° naphtha, filtering through dried fuller's earth and washing with additional warm 88° naphtha until the filtrate is clear. This removes the dark-colored asphaltic substances, which remain adsorbed by the fuller's earth. The filtrate containing the paraffin is distilled to a small bulk, evaporated to dryness on the water bath, under reduced pressure, the residue dissolved in 25 ml. of ether and 25 ml. of alcohol, and treated as described above.

Instead of removing the dark-colored asphaltic constituents by means of fuller's earth, an alternate procedure consists in extracting them by shaking the 88° naphtha solution three successive times with 20 ml. concentrated sulfuric acid in a separatory funnel followed with N. KOH (in 50 per cent alcohol-water solution) and finally with water until neutral to phenolphthalein.

Another method for separating paraffin wax from petroleum residues[200] consists in mixing the residue with an excess of fuller's earth, *cooling* and extracting either with: (1) *cold* benzol (which removes both the oily matters and the asphalt), or (2) *cold* gasoline (which removes the oily matters only) and then with *cold* benzol (which removes the asphalt). The residue of fuller's earth is then treated with *hot* water which separates the adsorbed paraffin wax. This procedure is precisely the reverse of the preceding (as well as the method described in Test 38*d*), which may however be explained by the fact that in this case the process is carried on *in the cold.*

The *refractive index* of the solid paraffins will indicate whether the original substance was ozokerite or paraffin, or a mixture of

the two. When tested at 90° C. on the Zeiss or Abbe refractometer, ozokerite will show a refractive index below 15.0, whereas the solid paraffins derived from petroleum, shale, lignite, etc., will test between 15.0 and 30.0.[201] When tested in the solid state, commercial paraffin waxes show a refractive index ranging from 1.500 to 1.540 at temperatures between 30 and 130° F.[202] Furthermore, paraffin wax has a lower molecular weight than ceresine or ozokerite—for melting points between 50 and 60° C., the former tests between 330 to 400, and the latter above 450.

SULFONATION RESIDUE

Test 34a. Residue Insoluble in Concentrated Sulfuric Acid. This method of test has been proposed for separating saturated and unsaturated hydrocarbons, as a means of distinguishing various asphaltic products (e.g., native asphalts and petroleum asphalts) from tars and pitches, thereby serving for purposes of identification. Two alternate procedures are available, viz:

(*a*) *Portion Soluble in* 88° *Petroleum Naphtha.*[203] The portion soluble in 88° petroleum naphtha, separated as in Test 23, is brought to exactly 100 ml., either by adding more 88° naphtha or else by evaporation, so that the quantity of substance carried in solution will be approximately 5 grams. This is then shaken in a 500 ml. separatory funnel at 77° F. for *exactly* three minutes, with 30 ml. of a mixture of concentrated sulfuric acid and fuming sulfuric acid, having a specific gravity of 1.84 at 77° F. The funnel is allowed to stand quietly overnight, whereupon the acid is drawn off and the oils unacted upon treated with another 30 ml. of the acid. This time a few hours' standing should effect a sharp separation. If the second acid layer is strongly colored, the treatment should be repeated a third time. The naphtha solution is washed successively with water, a 5 per cent solution of sodium carbonate and finally with water. The solution is evaporated to dryness over a steam bath and the residue weighed. This is equal to the saturated hydrocarbons present in the portion soluble in 88° petroleum naphtha. As a guide in evaporating the last traces of naphtha from the saturated hydrocarbons, a blank test should be run on 100 ml. of the 88° naphtha, whereupon the portion unacted upon is mixed with 0.75 g. of a non-asphaltic petroleum residuum and evaporated on the steam bath alongside of the sample under test, until the former is reduced to exactly its original weight.

The results are expressed as the percentage of saturated hydro-

carbons present in the portion soluble in carbon disulfide (Test 21). This is calculated in the following manner: If *a* represents the percentage soluble in carbon disulfide, *b* the percentage soluble in 88° naphtha and *c* the percentage of saturated hydrocarbons in *b*; then the saturated hydrocarbons present in the portion soluble in carbon disulfide will equal $\frac{bc}{a} \times 100$.

(*b*) *Distillate at 315–355° C.* This test expresses the percentage of saturated hydrocarbons in the distillate between 315 and 355° C. obtained upon subjecting the bituminous substance to the flask method of distillation (Test 16*b*). It is used to differentiate tars and pitches among themselves as well as from mineral waxes, asphalts (native and pyrogenous) and asphaltites. The figures for coal-tar pitches have already been given. The author cites the following additional figures: wood-tar pitch 0 per cent, saponifiable fatty-acid pitches 0 per cent, unsaponifiable fatty-acid pitches 0 per cent, residual asphalt from Mexican asphaltic petroleum 86 per cent, wurtzilite asphalt 87 per cent and gilsonite 85 per cent.

The method of determining the sulfonation residue as proposed by Dean and Bateman [204] consists in distilling sufficient of the material under examination by the flask method (Test 16*b*) to obtain at least 10 ml. of distillate between 315 and 355° C. Exactly 10 ml. of this fraction are measured into a Babcock milk bottle, and 40 ml. of 37 normal sulfuric acid added, 10 ml. at a time. The bottle and its contents are shaken for two minutes after each addition, and when all the acid has been added, the bottle is kept at a constant temperature of 98–100° C. for one hour, during which it is shaken vigorously every ten minutes. At the end of the hour, the bottle is removed, cooled, filled to the top of the graduations with ordinary sulfuric acid, and whirled for five minutes in a Babcock separator. The unsulfonated residue multiplied by 2 gives the per cent by volume directly (each graduation being equal to 1/200 of a ml.).

It is important that the acid should be of the proper strength. A mixture of fuming sulfuric acid and ordinary concentrated sulfuric acid should be prepared to contain exactly 80.07 per cent of SO_3, which constitutes 37 normal acid. If the sulfonation residue is dark in color, it should be treated with an excess of a 10 per cent sodium hydroxide solution, and if completely soluble in this reagent, the test is regarded as negative.

Test 34b. Residue Insoluble in Water.[205] Treat 3 g. of the carbon disulfide or benzol soluble portion of the substance, after evaporation of the solvent, with 6 ml. of concentrated sulfuric acid

(95 per cent) in a stout test-tube and agitate continuously for three-quarters of an hour while heating in boiling water. The contents of the test-tube are then poured into 500 ml. of cold water. Brown-coal tar and brown-coal-tar pitch are practically insoluble in cold water, and partly soluble in hot water. Coal-tar pitch, wood-tar pitch, oil-gas-tar pitch and naphthol pitch are completely soluble in cold water, forming a deep black solution, which upon filtering leave little to no residue. Lignite-tar pitch, peat-tar pitch, bone-tar pitch, rosin pitch, fatty-acid pitch, wool-fat pitch and montan pitch are partly soluble in cold water. Asphalts (both natural, as well as those derived from petroleum) and asphaltites are insoluble in cold water, forming a copious insoluble precipitate.[206] Let the solution stand for two hours and without agitating the precipitate, decant carefully through a weighed Gooch crucible of about 7 cm. diameter. Wash the residue with hot water, and finally transfer the precipitate to the filter. Continue washing with water until the filtrate no longer gives an acid reaction with methyl orange, then dry at 105° C. and weigh. Marcusson used this method for determining the percentage of coal-tar pitch in admixture with refined Trinidad asphalt. He found that upon treating the CS_2 soluble portion of this asphalt with sulfuric acid as outlined above, a small amount of soluble sulfo-derivatives was formed, so that an average correction of 4 per cent must be made to obtain accurate figures, as shown in Table CVII.

The foregoing test has been modified for the ostensible purpose of yielding more accurate results, as follows:[207] 2 g. of the substance are weighed in a test-tube 3.5 cm. in diameter, which is immersed 12 cm. in a 30 per cent solution of salt, maintained at 107–108° C., whereupon 20 g. of concentrated sulfuric acid (95 per cent) are added. A mechanical glass stirrer is introduced and caused to revolve at the rate of 300 turns per minute and the digestion continued for 2½ hours. The mechanical stirring is claimed to break up the lumps and result in a better filtration. Carefully introduce water in the test-tube, to a point 12 cm. from the bottom and continue the stirring for another 15 minutes, then transfer the contents of the tube into 1 liter of boiling water. Let stand overnight and carefully filter through two layers of tared filter paper in a 9 cm. porcelain filter, using suction. Wash the

TABLE CVII

SEPARATION OF MIXTURES OF TRINIDAD ASPHALT AND COAL-TAR PITCH

Crude Trinidad Asphalt (Containing 45% Soluble Constituents), Per Cent	Refined Trinidad Asphalt (Containing 58% Soluble Constituents), Per Cent	Coal-tar Pitch, Per Cent	CS_2 Soluble Constituents of the Asphalt Present in the Mixture			
			Per Cent Calculated	Per Cent Found	Per Cent Corrected (+4.0%)	Per Cent Difference
15	.....	85	6.8	4.2	8.2	+1.4
20	.....	80	9.0	6.4	10.4	+1.4
25	.....	75	11.3	8.4	12.4	+1.1
30	.....	70	13.5	11.0	15.0	+1.5
40	.....	60	18.0	13.4	17.4	−0.6
45	.....	55	20.3	14.6	18.6	−1.7
60	.....	40	27.0	22 8	26.8	−0.2
.....	20	80	11.6	7.3	11.3	−0.3
.....	23	77	13.4	9.2	13.2	−0.2
.....	30	70	17.4	12.3	16.3	−1.1
.....	35	65	20.3	14.9	18.9	−1.4
.....	40	60	23.2	16.9	20.9	−2.3

residue with boiling water until the filtrate is clear and no longer reacts acid with methyl orange. The filter containing the residue is placed on a Petri dish and dried for 2 hours at 105° C., cooled in a desiccator and weighed. This represents the asphaltic constituents present, which, however, are contaminated with a certain proportion of *insoluble* sulfo-derivatives of the asphalt. An examination of a range of petroleum asphalts, as well as Trinidad asphalt, indicated that the weight of insoluble asphaltic constituents with their associated insoluble sulfo-derivatives, as ascertained by this method must be corrected by deducting 20 per cent, to obtain the net weight of asphaltic constituents actually present. Similarly, an examination of a number of coal-tar pitches showed that in no case was more than 0.1 per cent of insoluble sulfo-derivatives present. No correction need therefore be made for pitches. Any free carbon present in the pitch (ascertained by the insoluble matter separated upon originally dissolving the substance in carbon disulfide) should be taken into account by using the following formula:

$$\text{Per cent asphalt} = 80\%\ (100 - C) \cdot \frac{S}{E}$$

where: C = per cent free carbon present in the mixture; S = grams residue obtained after sulfonation; E = gram mixture (after having been freed from insoluble constituents and carbonaceous matter) taken for the sulfonation process.

Heinrich Mallison claims [208] that asphalts are converted into both soluble and insoluble sulfo-derivatives, and that certain asphalts when sulfonated by themselves showed slight increase in weight, whereas when sulfonated in admixture with 85 per cent of coal tar, they were converted almost quantitatively into soluble products.

Test 34c. Dimethyl Sulfate Method. This was originally proposed as a qualitative method for detecting mixtures of pitches with asphalts. It is based upon the fact unsaturated hydrocarbons are soluble in dimethyl sulfate, whereas saturated hydrocarbons are not. The material is distilled as in the preceding test, and 4 ml. of the distillate shaken with 6 ml. of dimethyl sulfate in a 10 ml. cylinder graduated to 0.1 ml. After standing, the percentage of insoluble hydrocarbons (i.e., saturated) is read off.[209] The results are only approximate. Diethyl sulfate has also been proposed for the purpose, and is claimed to be more reliable.[210]

FORMOLITE REACTION

Test 35. Nastjukoff Method. This is based upon the observed fact that cyclic unsaturated hydrocarbons, resins and asphaltic constituents yield insoluble formolite, whereas paraffins, olefins, naphthenes, polynapthenes, ketones and sulfur compounds remain unchanged.[211]

The test is performed as follows: Dissolve ½ to 1 g. of the substance in 35 ml. carbon tetrachloride, cool in an ice-bath and add 15 ml. concentrated sulfuric acid, drop by drop, with constant agitation. While still on the ice-bath, add 5 ml. of 40 per cent formaldehyde, drop by drop, and allow the mixture to stand for fifteen minutes. Dilute upon cooling with 100 ml. water and neutralize with strong ammonia. After standing fifteen minutes, decant the liquor through a tared Gooch crucible. Wash the residue in the flask with water and then with CCl_4, and pour the washings through the filter. The contents of the flask are then washed into the filter with water and CCl_4, and washed alternately with water and CCl_4

until the washings run through clear. The residue is then dried in an air-bath at 220° F. for one hour, cooled and weighed. The weight of the residue is calculated in percentage of the original sample, and represents the "formolite residue." It has been found that upon blowing asphalts, there is an increase in the amount of formolite residue.[212]

DEGREE OF MERCURATION

Test 36. Conventional Method. This is based upon the fact that mercuric acetate reacts with unsaturated hydrocarbons and sulphur compounds, accompanied by the liberation of acetic acid.[213] A benzol solution of the bituminous substance is shaken with a solution of $Hg(OAc)_2$ in methyl alcohol, and the liberated acetic acid is estimated by titration. The number of milliliters of N/KOH required to neutralize the acid liberated by 100 g. of the substance is termed the "degree of mercuration." A solution of mercuric nitrate (Millon's reagent) has been proposed,[214] to detect coal-tar products in admixture with asphalt. This is accomplished by boiling 10 g. of the substance with 25 ml. N/NaOH for 20 minutes, filtering and adding nitric acid until almost neutral. Then concentrate to 5–10 ml., add 5 ml. of Millon's reagent and heat the mixture in a test-tube in boiling water for 30 minutes. The presence of even 1 to 2 per cent of coal tar or coal-tar pitch will cause a coloration, whereas no color will develop with asphalt or rosin.

SAPONIFIABLE CONSTITUENTS

In the case of bituminous materials, the estimation of the unsaponifiable and saponifiable matters is of value for purposes of identification. Certain bituminous substances, such as montan wax, rosin pitch, and fatty-acid pitch are often composed largely of saponifiable constituents. Others, including pine-tar, pine-tar pitch, hardwood tar, hardwood-tar pitch, peat tar, lignite tar, bone tar, bone-tar pitch and other forms of fatty-acid pitches contain smaller percentages. This test is also used for gauging the uniformity of supply, and in the case of fatty-acid pitches, as a criterion of the quality.

The relation between the acid, lactone, ester and saponification

values, also the unsaponifiable and saponifiable constituents is shown in Table CVIII.

Test 37a. Free Acids ("Acid Value"). Boil 5.00 g. of the material under a reflex condenser with 25 ml. of benzol until the material dissolves, or else is completely disintegrated, then add 100 ml. of carefully neutralized 95 per cent ethyl alcohol and continue boiling for twenty minutes.* The liquid is decanted from the insoluble residue while hot, the latter boiled with another 50 ml. of alcohol, and the process repeated, until the extract no longer

TABLE CVIII

Saponification Value (Saponifiable Matter)	Acid Value	Free Fatty Acids	
		Free Resin Acids	
		Free Asphaltous Acids	
	Lactone Value	Anhydrides and Lactones	
	Ester Value	Neutral Fats	[Glycerol]
			Fatty Acids
		Waxes	Fatty Acids
			Higher Alcohols
Unsaponifiable Matter		Free Higher Alcohols	
		Hydrocarbons	

reacts acid with alkali blue 6-B (or phenolphthalein). The residue is then disregarded. To the combined extracts, add 10 ml. of a 25 per cent barium chloride solution and 6 drops of a 3 per cent alcoholic solution of alkali blue 6-B (or an equivalent amount of 1 per cent alcoholic phenolphthalein), and titrate *cold* with standard N/10 caustic potash.† As the free acids are neutralized by the alkali, the barium soaps are precipitated, and at the same time the unsaponified substances are thrown out by the water contained in the N/10 caustic potash, until at the close of the titration the solu-

* Bituminous materials with high fusing-points should be fluxed to semi-liquid consistency with a given weight of neutral paraffin oil.

† Prepared by dissolving 5.612 g. pure caustic potash in 500 ml. 95 per cent alcohol, diluting to exactly 1 liter with water at 60° F. and carefully standardizing against sulfuric acid of known strength.

tion becomes almost clear, rendering the end-point sharp. The acid value is equivalent to the number of milligrams of potassium hydroxide required to neutralize the free acids in 1 g. of the substance.[215]

Test 37b. Lactones and Anhydrides ("Lactone Value"). These are determined as follows: (1) Find the acid value (Test 37*a*), and the ester value (Test 37*c*) of the original substance. (2) Find the acid value (Test 37*a*) and the ester value (Test 37*c*) of a weighed quantity of the free acids liberated from the substance after saponification. If acid and ester values (1) are equal to respective acid and ester values (2); then lactones *only* are present. If acid value (1) is less than acid value (2), and ester value (2) is equal to 0; then glycerides *only* are present. If acid value (1) is less than acid value (2), and ester value (1) is greater than ester value (2); then *both* glycerides and lactones are present.

The true ester value is equal to ester value (1) minus ester value (2); and the true lactone value is equal to ester value (2).

The foregoing results may be checked by finding the acid values of the original substance and the liberated acids. The true ester value equals the acid value of the free acids minus the acid value of the original material. Similarly, the lactone value is equal to the saponification value minus the sum of the acid and ester values.

Test 37c. Neutral Fats ("Ester Value"). The ester value corresponds to the number of milligrams of potassium hydroxide consumed in saponifying esters (neutral fats, otherwise known as triglycerides). If lactones or anhydrides are absent, the ester value may be *calculated* by subtracting the acid value from the saponification value. If lactones and anhydrides are present, then the ester value may be calculated by subtracting the sum of the acid and lactone values from the saponification value.

Test 37d. Saponification Value. The saponification value represents the number of milligrams of potassium hydroxide consumed in the *complete* saponification of 1 g. of the substance. It represents the sum of the acid, lactone and ester values, and is ascertained in the following manner:

Prepare a 5 per cent solution of caustic potash dissolved in equal volumes of 95 per cent ethyl alcohol and 90 per cent thiophene-free benzol or xylol, let stand at least twenty hours, filter through an

asbestos filter and standardize against sulfuric acid of known strength.* Saponify 5 g. of the substance† with 50 ml. of this solution (added from a calibrated pipette) by boiling over an electric hot plate under a reflux condenser 1 to 4 hours, depending upon the rapidity with which the substance goes into solution. Before disconnecting the flask, wash out the condenser with a few milliliters of neutral alcohol. Evaporate the benzol on a water bath, add 100 ml. water, boil, decant from the residue, add 50 ml. more water, boil, decant and repeat until all the alkali has been removed (tested by adding a drop of phenolphthalein). Combine the extracts, add 20 ml. of 25 per cent barium chloride solution ($BaCl_2 \cdot 2H_2O$), and 3 ml. each of 1 per cent alcoholic phenolphthalein solution and a 3 per cent alcoholic solution of alkali blue 6-B.‡ Titrate the warm solution with normal sulfuric acid. As the barium hydroxide becomes neutralized, a copious precipitate of barium sulfate forms which renders the end-point distinct. When the color changes, boil, and if necessary run in more sulfuric acid until the color remains green on boiling. Calculate the quantity of caustic potash required for saponification.[216]

The foregoing method may be used for dark-colored, difficultly saponifiable substances, such as montan wax. The use of normal butyl alcohol in place of ethyl alcohol will enable the saponification to proceed more rapidly, on account of its higher boiling point, and is therefore recommended for substances which saponify with difficulty.[217]

A potentiometric method has been proposed[218] for ascertaining the saponification value of asphalts and mixtures of asphalts with vegetable oils, consisting of the following: To 1 g. of the substance add 50 ml. of approximately 7/10 N. KOH in ethyl alcohol and 40 ml. c.p. anisole (methylphenylester). Boil under a reflux condenser for one hour, cool and titrate potentiometrically with standard aqueous HCl. A normal calomel electrode and a hydro-

* Approximately 45 ml. of N. sulfuric acid will be required to neutralize 50 ml. of the 5 per cent caustic potash solution.

† If the substance fuses above 180° F. (R. and B. Method), flux it back to this fusing-point, or lower, with a neutral mineral oil, which should previously have been run for a blank test.

‡ Julius Marcusson finds that by using the two indicators together, the end-point of the titration is sharper, being evidenced by a change in color from red to green.

gen electrode of the usual type are used as reference and indicator electrodes respectively. During the titration two layers will separate, the upper anisole layer containing almost all the asphalt. About 9 minutes are required to attain a steady potential after each addition of acid. The first maximum represents the neutralization of excess alkali, and the last maximum represents the completion of the reaction between the HCl and the alkali in combination. Hence the amount of acid used between the first and last maxima is a direct measure of the combined alkali. By this method, the saponification value of gilsonite was found to be 9.9 and that of Bermudez asphalt 27.5. The foregoing procedure is particularly suitable for mixtures having a dark color, where readings with indicators are difficult to obtain.

Another modification [219] consists in dissolving the substance in 1 to 2 parts by weight of white petroleum oil and shaking with anhydrous alcoholic KOH for 30 to 60 minutes at 100° C. On cooling to room temperature without agitation, the saponified residue will adhere to the bottom of the flask, whereupon the alcoholic layer containing some of the oil is poured off for titration.

Test 37e. Separation of Saponifiable Constituents. The following procedure has been devised by the author specifically for examining bituminous materials or admixtures of bituminous materials with animal or vegetable oils and fats, since the customary methods do not adapt themselves especially well, due to the formation of troublesome emulsions. The bituminous material is first freed from insoluble constituents, including any mineral matter, by boiling with carbon disulfide under a reflux condenser, cooling and filtering through a Gooch crucible, following the precautions described in Test 21. The insoluble constituents are dried at 100° C. and weighed. Sufficient of the bituminous substance should be taken to yield approximately 5.0 g. of extract. The benzol solution is evaporated or distilled to 50 ml., and 50 ml. of the saponifying liquid added from a pipette. This should consist of a 10 per cent solution of caustic potash, prepared by dissolving 100 g. of anhydrous potash in 500 ml. of 95 per cent ethyl alcohol, and diluting to a liter with 90 per cent benzol. The liquid is allowed to stand overnight to permit any carbonate to settle, and the clear solution decanted. After the saponifying agent is added, the mixture is boiled

under a reflux condenser for one-half to one hour, and the contents of the flask, while still warm, poured in a separatory funnel containing 150 ml. of boiling water and 25 ml. of a 10 per cent solution of potassium chloride. Add 250 ml. of benzol, agitate vigorously, and allow the funnel to rest quietly in a warm place until the solvent separates. If an emulsion forms which refuses to separate on standing, add 200 ml. more benzol and 100 ml. 95 per cent ethyl alcohol and stand in a warm place overnight. This will invariably effect a more or less complete separation of the solvent. From this point on the method is illustrated by the following tabular outline:

Saponify as described:
Draw off the soap solution as completely as possible.
Decant the benzol layer, leaving the intermediate layer in the separatory funnel.

Aqueous Soap Solution. Exhaust with 200-ml. portions of benzol.	Benzol Layer.	Intermediate Layer.
Aqueous Soap Solution.	Combined benzol Extracts.	
Combine and exhaust with 100-ml portions of 50 per cent alcohol.		
Benzol Solution.	Combined Alcoholic Extracts.	
	Combine and exhaust with benzol.	
	Combined Benzol Extracts.	Alcoholic Soap Solution.
Combine, evaporate to a small bulk, complete the evaporation at 100° C., cool and weigh the *Unsaponifiable Constituents.*		
Combine		

Transpose with dilute hydrochloric acid, warm and exhaust with benzol. Separate the aqueous solution containing the glycerol and mineral salts. Evaporate the combined benzol extracts to a small bulk, and then complete the evaporation of solvent at 100° C. Cool and weigh. Weight equals the *free acids derived from the saponifiable constituents.*

In the case of bituminous materials that are more or less completely saponifiable, the intermediate layer is apt to be absent. In this case the process will simplify itself considerably. The foregoing procedure will separate the unsaponifiable constituents in practically an ash-free state.[220]

Test 37f. Examination of Unsaponifiable Constituents. To separate the hydrocarbons, boil 2 g. of the unsaponifiable matter with 4 ml. of acetic anhydride under a reflux condenser for one hour. Add 25 ml. of 95 per cent ethyl alcohol, heat to boiling, decant through an asbestos Gooch crucible, and remove all traces of acetic anhydride by washing with successive portions of warm alcohol. Dry the residue on the Gooch at 100° C. Its weight is equal to the hydrocarbons present.

To separate the higher alcohols (cholesterol), the filtrate from the foregoing is evaporated to dryness, then dissolved in the smallest possible quantity of hot absolute ethyl alcohol and allowed to cool. The cholesterol and phytosteryl (sometimes termed sitosteryl) will crystallize as acetates. Filter and wash with 95 per cent alcohol. Find the melting-point by the capillary-tube method as ordinarily used for pure organic substance. Cholesterol acetate will melt between 114.3 and 114.8° C., whereas phytosteryl acetate will melt above 125° C. Recrystallize several times from hot absolute alcohol and redetermine the melting-point. If the fifth to seventh crop of crystals test below 115–116° C., then phytosteryl is absent.

Cholesterol may also be detected by boiling 1 g. of the substance with 2 ml. of chloroform and 20 drops of acetic anhydride. The solution is allowed to cool and the clear liquid decanted into a porcelain crucible. Then 1 drop of concentrated sulfuric acid is added to the liquid. If cholesterol is present, a violent-pink to reddish coloration will be obtained. (For the behavior of resin acids in the foregoing test, see Test 41.)

Cholesterol indicates the presence of *animal* oils, fats or waxes (such as wool grease), whereas phytosteryl indicates *vegetable* oils, fats or waxes. This test is therefore of value in detecting which class of substances is present in admixture with bituminous material.

Test 37g. Examination of Saponifiable Constituents. If both fatty and resin acids are present, these may be separated quantitatively by the Twitchell-Gladding method, or by the Hans Wolff method, the details of which will be found in standard textbooks.

Test 37h. Glycerol. The presence of animal and vegetable oils or fats (triglycerides) will be indicated by determining the glycerol liberated upon saponification. Certain fatty-acid pitches similarly contain a small percentage of glycerol. This is determined as fol-

lows: Saponify 5–10 g. of the substance under examination, weighing exactly, and using 25 ml. of the saponifying agents described in Test 37*e*. Extract the unsaponifiable constituents with benzol as described, and then transpose the soap solution with a *slight* excess of dilute sulfuric acid (instead of using hydrochloric acid). Warm the liquid and extract the fatty acids with benzol.

Evaporate the aqueous solution to a small bulk, and make slightly alkaline with dilute caustic soda. Cool, dilute to about 100 ml. and determine glycerol by any of the standard methods proposed for this purpose.[221]

ASPHALTIC CONSTITUENTS

The methods which follow have been proposed by Julius Marcusson [222] for differentiating between native and petroleum asphalts. They also give an insight into the composition of asphalts themselves, and in this respect the author regards them of special merit. The value and possibilities of these determinations do not appear to be generally appreciated, but as time goes on they will certainly be recognized. A schematic outline of the procedure is shown in Table CIX, in which is included a further refinement proposed by Hans Pöll [223] for separating the asphaltenes into its two components. Table CX gives the results reported from several sources [224] applicable to typical asphalts.

Table CXI shows the effect of the blowing process on a specimen of petroleum asphalt.[225] It will be observed that the percentage of asphaltenes is increased at the expense of the asphaltic resins and oily constituents. In addition, the particles of carbon coalesce through the abstraction of the protective bodies and become coarser, as is evidenced under the ultramicroscope. The higher asphaltene content results in a higher fusing-point; the decreased content of asphaltic resins decreases the ductility; and the high content of oily constituents accounts for a low break-point, resulting in the blown product having a great elasticity at low temperatures, together with pronounced rubber-like characteristics.

Test 38a. Free Asphaltous Acids. Dissolve 5.00 g. of the asphalt in 25 ml. benzol by boiling under a reflux condenser. Add 200 ml. of neutral ethyl alcohol, let settle, decant the solution from the pitchy residue, and titrate the former cold with N/10 alcoholic

TABLE CIX—SCHEMATIC OUTLINE OF PROCEDURE TEST 38

Dissolve 5 g. asphalt in 25 ml. c. p. benzol under a reflux condenser with boiling. Add 200 ml. neutral ethyl alcohol, let settle and decant.

Alcoholic Extract:
Neutralize with N/10 alcoholic NaOH, using phenolphthalein as indicator; dilute with an equal volume of water; extract the unsaponified constituents with benzol.

Residue:

Alcoholic Soap Solution:
Evaporate to a small bulk; acidify with HCl; and extract with benzol.

Benzol Extract:
↑ Combine ↑
Saponify by boiling 1 hour under a reflux condenser with N/1 alcoholic NaOH in the presence of benzol; dilute with an equal volume of water; extract the unsaponified constituents with benzol.

Benzol Extract:
Evaporate to dryness at 105° and weigh:
Free Asphaltous Acids

Alcoholic Soap Solution:
Evaporate to small bulk; acidify with HCl; extract with benzol.

Benzol Extract:
Evaporate to dryness at 105° and weigh:
Asphaltous Acid Anhydrides

Unsaponified Residue (Note A):
Dissolve in small quantity (±10 ml.) benzol; pour into 200 ml. 88° petroleum naphtha; let settle for several days; filter through a Gooch crucible; wash with 88° petroleum naphtha.

Residue:
Dry at 105° C. and weigh:
Asphaltenes

Asphaltenes (Note B):
Dissolve in $CHCl_3$; distribute over 25 g. fullers' earth in a paper thimble; extract with successive portions of cold pyridine.

Residue on Fullers' Earth:
Extract with a mixture of pyridine and CS_2 (1 : 1); distil off the CS_2 and evaporate to a small bulk under vacuum in a stream of CO_2. Add hot water to separate the asphaltic constituents; filter and wash with hot water until free from pyridine. Dissolve in $CHCl_3$; evaporate to dryness at 105° C and weigh.
Hard Asphalt Resins in Asphaltenes

Pyridine Extract:
Evaporate the combined extracts to a small bulk under vacuum in a stream of CO_2. Add $CHCl_3$, and wash with HCl to remove all traces of pyridine. Evaporate to dryness under vacuum at 100° C. in a stream of CO_2 and weigh.
Hard Asphaltic "Carboids" in Asphaltenes

88° Petroleum Naphtha Solution:
Evaporate to 25 ml.; distribute over 25 g. fullers' earth in a paper thimble; extract in Soxhlet with hot 88° petroleum naphtha.

Residue on Fullers' Earth:
Extract with CS_2; evaporate to dryness at 105° C and weigh.
Asphaltic Resins

88° Petroleum Naphtha Extract:
Distil to small bulk; evaporate to dryness at 100° C. under vacuum and weigh.
Oily Constituents

Note A—To ascertain only the asphaltenes, asphaltic resins and oily constituents, start the test at this point, using 5 g. of the mineral-free asphalt.

Note B—The separation of asphaltenes into "Hard Asphaltic Carboids" and "Hard Asphaltic Resins" constitutes an elaboration proposed by Hans Pöll.

TABLE CX

Asphaltic Constituents Present in Typical Asphalts

Type of Asphalt:	Fusing-Point °F.	Penetration 100 g/5 sec. At 77° F.	Penetration 100 g/5 sec. At 100° F.	Ductility at 77° F.	Break-point °F.	Free Asphaltous Acids, %	Asphaltous Acid Anhydrides, %	Asphaltenes, %	Asphaltic Resins, %	Oily Constituents, %
Natural Asphalts:										
Alberta asphalt sand(*a*)	50(*c*)	Soft	Soft	7	.....	2 0		22.5	24.0	51.5
Val de Travers asphalt(*a*)	50(*c*)	Soft	Soft	.....	.....	7 1	1 3	12.9	33.2	42.2
Trinidad asphalt(*a*)	132(*c*)	4	.....	9	. . .	6 4	3.9	37.0	23.0	31.0
Bermudez asphalt (refined)	135(*c*)	25	.....	11	...	3 8	2 0	35.3	14.4	39.6
Fluxed grahamite(*b*)	161(*c*)	.....	.....	.. .	.. ..	0.90	1 28	17.28	30.75	48 50
Residual Oils:										
U. S. Mid-continental petroleum	80(*c*)	Soft	Soft	Soft	....	0.92	0.46	Trace	25.34	74.59
Californian petroleum	81(*d*)	Soft	Soft	Soft	.....	... .	.....	4.04	34.41	60 71
Mexican (Panuco) petroleum	85(*d*)	Soft	Soft	Soft	....	.....	.. ...	13 65	26 64	59 20
Cracking-coil (low pressure)	103(*d*)	183	Soft	+110	.....	.....	.....	11.56	13.92	74.52
Cracking-coil (high pressure)	105(*d*)	150	Soft	+110	.....	.....	.....	14.43	9.32	76 15
Blown Petroleum Asphalts:										
Illinois petroleum	133(*d*)	30	93	27	.....	.....	.....	18 32	28.30	52.80
Venezuelan petroleum	140(*d*)	60	.....	12	−18½	.....	.....	20 0	26.0	54 0
Californian petroleum	144(*d*)	21	70	31	.....	.....	.....	19.16	28 54	52 30
Mexican (Panuco) petroleum	144½(*d*)	40	94	12	.....	.....	.....	16.38	29 93	53.69
Colombian petroleum	147½(*d*)	39	92	14	.....	.....	.....	13.66	36.76	49.58
Venezuelan (Quire Quire) petr'm.	148(*d*)	16	47	40	.....	.....	.....	26 97	4.45	68 58
Cracking-coil (low pressure)	148(*d*)	40	100	14	.....	.....	.....	26.84	28.26	44 88
Cracking-coil (high pressure)	148(*d*)	22½	63	25	.....	.....	.....	22.75	8.24	69 01
Mexican (Panuco) petroleum	155(*d*)	42	91	10	.....	.....	.....	30.25	27 30	41.83
Mexican petroleum	192(*d*)	19	5	1½	.....	.....	.....	30.4	17.6	52 0
Residual Asphalts:										
Kansas petroleum	.....	.....	.....	.....	.....	0.0	3.0	24.0	11.0	62 0
Russian petroleum	.....	... ..	.....	.....	. ....	0.0	2.0	15.5	16 1	66 0
German petroleum	.....	.....	.....	.....	.....	0.0	4.0	4.4	8.6	83.0
Illinois petroleum	102(*d*)	235	Soft	+110	.....	.....	.....	5.24	35.12	59 49
Venezuean (Quire Quire) petr'm.	102½(*d*)	190	Soft	+110	.....	.....	.....	3.19	23.88	70.65
Colombian petroleum	107½(*d*)	189	Soft	+110	.....	.....	.....	6.90	20.40	72 60
Mexican (Panuco) petroleum	108(*d*)	160	Soft	+110	.....	.....	.....	16.40	30 77	52 83
Californian petroleum	131(*d*)	39	.....	+100	26½	.....	.....	4.6	59.0	36.4
Venezuelan petroleum	133(*d*)	34	.....	+100	7	.....	.....	16.0	35.0	49.0
Mexican petroleum	134½(*d*)	38	.....	76	10½	.....	.....	22 0	34.0	44.0
Mexican petroleum	160(*c*)	.....	.....	.....	.....	0 61	Trace	5.81	26 72	65.45
Sludge Asphalt:										
U. S. petroleum (blended)	180(*c*)	.....	.....	.....	.....	0.81	1.61	27.01	25.68	44.09

(*a*) Freed from associated mineral constituents.
(*b*) Grahamite 15% fluxed with residual oil (from asphaltic petroleum) 85%.
(*c*) K. and S. method
(*d*) R. and B method.

TABLE CXI

EFFECT OF THE BLOWING PROCESS ON THE ASPHALTIC CONSTITUENTS

	Fusing-point (K. & S.) °F.	Asphaltous Acids and Anhydrides %	Asphaltenes %	Asphaltic Resins %	Oily Constituents %	Ash %	Total %	Molecular Weight of Asphaltenes	Fusing-point Asphaltenes °F.
Original asphalt	93	1 85	24 2	15 0	55.0	4.3	100 35	2219	313–324
Blown asphalt	219	0.87	47.8	9 3	37 8	4.5	100 27	2560	491–505
" "	272	0 64	48 4	8 4	39 2	4 2	100 84	3130	579–587
" "	304	0 46	53 4	7 8	34 9	4.62	101 18	4225	635–644
" "	336	0.42	57 8	8.0	29 1	4.38	99 70	4690	680–695

sodium hydroxide, using phenolphthalein as indicator. Dilute with an equal volume of water and extract the unsaponifiable constituents by shaking with successive portions of benzol until the extract becomes clear. Evaporate the alcoholic soap solution to a small bulk, liberate the asphaltous acids by acidifying with hydrochloric acid, extract with benzol, evaporate the extract to dryness at 105° C. and weigh. The free asphaltous acids appear as a brownish-black tar-like to resinous mass, soluble in alcohol, benzol and chloroform, but nearly insoluble in 88° petroleum naphtha. When heated to 120–200° C. they are converted into the corresponding anhydrides, and at higher temperatures are transposed into unsaponifiable substances resembling asphaltenes. In ethereal solutions they are precipitated by mercuric bromide. With vanillin-hydrochloric acid they give the same color reaction as aliphatic ketones.[226]

Test 38b. Asphaltous Acid Anhydrides. In the foregoing test, the unsaponified portion is united with the pitchy substances precipitated by alcohol from the original benzol-alcohol solution. These are saponified by boiling under a reflux condenser for one hour with N-alcoholic caustic potash in the presence of benzol, the solution is diluted with an equal volume of water, and the unsaponified constituents extracted with successive portions of benzol. The alcoholic soap solution is then evaporated to a small bulk, the asphaltous acid anhydrides liberated by acidifying with hydrochloric acid, extracted with benzol, evaporated to dryness at 105° C. and weighed. These are very similar in appearance to the free asphaltous acids. On heating to high temperatures, they are converted into unsaponifiable products similar in appearance to the asphaltenes.

Test 38c. Asphaltenes. After separating the saponifiable constituents according to Tests 38*a* and 38*b*, the bodies which have not combined with alkali are dissolved in the smallest possible quantity of benzol (not exceeding 10 ml.), and the solution poured into 200 ml. of 88° petroleum naphtha.* The insoluble matter is filtered on a Gooch crucible as described in Test 21, washed with 88° naphtha, dried and weighed. This represents the asphaltenes, which appear as a dark brown to black powder similar to grahamite in characteristics. On heating, asphaltenes do not melt, but swell and decompose into a compact and hard coke. Asphaltenes are supposed to be formed by the addition of oxygen or sulfur to petroleum resins, also by intermolecular changes taking place on heating them in air. They are soluble in benzol, chloroform and carbon disulfide, almost insoluble in alcohol and 88° petroleum naphtha, and sparingly soluble in ether and acetone. The asphaltenes derived from natural asphalts are characterized by the presence of oxygen and a high percentage of sulfur (7 to 13 per cent), and under the influence of light are converted into an insoluble modification. The ratio of carbon to hydrogen is highest in the asphaltenes, diminishing in the remaining constituents.

The asphaltenes and their parent substances, the asphaltic resins, are regarded as saturated polycyclic compounds containing sulfur or oxygen, either of which can replace the other.

Asphaltenes are produced by heating the asphaltic resins, accompanied by a darkening in color and a gradually decreasing solubility in petroleum naphtha. Their specific gravity is greater than unity. The asphaltenes impart hardness to the asphalt, hence the greater the percentage present, the harder will be the asphalt. Asphaltenes and asphaltic resins (Test 38*d*) react with fuming nitric acid forming nitro-addition products, soluble in alcohol and acetone, and forming water-soluble products with alcoholic solutions of alkalies. Concentrated sulfuric acid and fuming sulfuric acid when heated, form addition products which are unaffected by alkalies. They also react with mercuric chloride and ferric chloride in chloroform solutions, forming double salts.

Test 38d. Asphaltic Resins. The solution of 88° petroleum naphtha obtained from Test 38*c* is evaporated to about 25 ml., dis-

* Of which at least 85 per cent by volume should distil between 35 and 65° C.

tributed over 25 g. fuller's earth in a paper thimble, and extracted hot in a Soxhlet with 88° petroleum naphtha. If the first extract is dark colored, it is concentrated to about 25 ml., poured over more fuller's earth, and the process repeated. The extract should have a straw or light yellow color. The asphaltic resins are adsorbed by the fuller's earth, from which they may be extracted by carbon disulfide, evaporated to dryness at 105° C., and weighed. These form the first stage in the conversion of petroleum hydrocarbons into asphaltenes, and consist of solid, reddish-brown to brownish-black substances fusing below 100° C., completely soluble in 88° naphtha, chloroform, carbon disulfide, benzol, but only sparingly soluble in hot or cold acetone. When adsorbed by fuller's earth they become insoluble in 88° petroleum naphtha and are no longer soluble therein after removal from the fuller's earth (probably due to oxidation by air while in contact with the earth). The asphaltic resins are formed by heating the oily constituents for some time to 120° C. accompanied by a darkening in color and absorption of atmospheric oxygen. High content of asphaltic resins produces great ductility and high break-point of the asphalt.

Sulfur dissolves in the resins when heated, with the liberation of hydrogen sulfide. The resins derived from coal-tar pitches when heated with strong sulfuric acid form water-soluble sulfo-compounds, whereas petroleum resins form insoluble products. Their specific gravity is in the neighborhood of unity.

Test 38e. Oily Constituents. The 88° petroleum naphtha extract from the fuller's earth in Test 38d, is distilled to a small bulk, and evaporated to dryness at 100° C. under vacuum until the odor of petroleum naphtha is no longer apparent. The oily constituents remaining as residue are weighed. These appear as a viscous oil, and constitute the most inert bodies contained in asphalts. They are optically active and usually fluorescent, show a specific gravity of less than unity, and have about the same iodine value as viscous lubricating oils derived from petroleum. In general they may be said to be composed of saturated and unsaturated hydrocarbons containing cyclic compounds, and containing small percentages of sulfur, nitrogen and oxygen.

As a general rule, the softer the asphalt, the larger will be the percentage of oily constituents. Marcusson reports that Trinidad

petroleum contains 42.5 per cent of oily constituents, Trinidad asphalt 17 to 19 per cent (figured on the crude dry substance containing the mineral ingredients) and grahamite 2 to 3 per cent.

The oily constituents derived from natural asphalts are characterized by the presence of small percentages of solid paraffins (usually less than 1 per cent) and by the fact that they appear fluid and sticky at 20° C., whereas those derived from petroleum asphalts appear non-fluid and of a consistency of vaseline. They impart softness to the asphalt, thus counteracting the hardening properties of the asphaltenes.

DIAZO REACTION

This test is used for identifying bituminous substances carrying phenols, including wood tar and wood-tar pitch, shale tar, peat and and lignite tars and pitches, bone tar, bone-tar pitch and the various coal-tar pitches. Oil-gas and water-gas tars and pitches contain but a trace of phenols and hence give but a slight diazo reaction.

Test 39. Graefe's Method. This reaction as devised by Edmund Graefe.[227] It is carried out by boiling 2 g. of the bituminous substance with 20 ml. N. aqueous caustic soda, for approximately five minutes. After cooling, the liquid is filtered. If the filtrate is dark colored, it may be lightened by shaking with finely pulverized sodium chloride and filtering. It is then cooled in ice to 10° C., and a few drops of freshly prepared diazobenzolchloride solution added (prepared by dissolving 1 g. aniline chlorhydrate in 10 ml. water and 3 ml. 25 per cent hydrochloric acid, and then adding drop by drop, a saturated solution of 0.5 g. sodium nitrite in water). To avoid the preparation of diazobenzolchloride (which does not keep), the use of paradiazobenzil-sulfonic acid ($C_6H_4 \cdot N_2SO_3$) may may be substituted, since it is a more stable chemical and will keep well in stock. If phenols are present, a more or less fugitive red coloration will result, sometimes accompanied by a reddish precipitate.

Assuming that the bituminous substance gives the diazo reaction, the question will often arise whether the product is a straight-distilled pitch, or an asphalt "cut-back" with a high boiling-point distillate containing phenolic bodies, derived from coal tar, lignite tar, etc. Julius Marcusson has worked out a method applicable under these circumstances,[228] which consists in dissolving 10 g. of the bitu-

minous substance in 15 ml. of benzol, and pouring the solution into 200 ml. of 88° petroleum naphtha. The resulting precipitate is filtered and washed with petroleum naphtha and dried. It is then boiled for fifteen minutes with N/2 alcoholic caustic potash under a reflux condenser to extract the phenols. The liquid is cooled and filtered, the alcohol evaporated, and the residue dissolved in water. Sodium chloride is added to clarify the liquid and remove any substances imparting a dark color, the solution is filtered and the filtrate treated for the diazo test described above. If a straight-distilled pitch containing phenols is present, a positive reaction will be obtained. If the original substance gives the diazo test, but the residue treated in the above way does not, then the admixture of high boiling-point oils containing phenolic bodies with a substance free from phenols (e.g., asphalts, etc.) is established. It is claimed that the presence of as little as 10 per cent of pitch containing phenols may be detected in this manner.

Where bituminous substances contain calcium carbonate, the phenolic bodies present combine with the lime, forming insoluble calcium phenolate which yields but a faint diazo reaction. However, on treating such substances with a solvent in the presence of hydrochloric acid, the calcium phenolate is decomposed, and the diazo reaction becomes much more delicate.

A still more sensitive test for ascertaining the presence of phenols (e.g., tars or pitches) in asphalts consists in the following: A reagent is prepared by dissolving 0.2 g. paranitraniline in 20 ml. water and 5 ml. 20 per cent sulfuric acid, whereupon 0.3 g. sodium nitrite are added. Hard asphalts (3 to 4 g.) are ground in a mortar with 25 ml. water made alkaline with NaOH; then filtered and the filtrate acidified with H_2SO_4 and 3 drops of the foregoing reagent added. The solution is finally made alkaline with NaOH, whereupon the presence of phenols will develop a more or less intense red coloration. It is claimed that the presence of 1 per cent of tar or pitch may thus be detected, inasmuch as a 1 : 25,000 solution of phenol will produce a blood-red color, and a 1 : 250,000 solution a pinkish-red color. Pure asphalts, on the other hand, yield a light yellow color or leave the reagent unchanged.

The Dutch Standardization Committee [229] have adopted a combination of paradiazobenzol-sulfonic acid with Millon's reagent for

detecting the presence of phenols in substances containing colophonium, as follows: 10 g. of the substance are boiled 20 minutes with 25 ml. N. aqueous NaOH and filtered. To 5 ml. of the filtrate add a few drops of paradiazobenzol-sulfonic acid. If no red coloration results, phenol (tar or pitch) is absent, but if a coloration occurs, tar or pitch may be present, but it may also be caused by colophonium. In this event, the remainder of the filtrate is almost neutralized with nitric acid and evaporated to 5 ml. Then add 5 ml. freshly prepared Millon's reagent and place in a glass beaker with boiling water. If no coloration results within half an hour, the mixture is free from phenols.

ANTHRAQUINONE REACTION

The anthraquinone reaction is used for detecting anthracene in tar products produced at high temperatures, including oil-gas tar and pitch, water-gas tar and pitch, and the various coal-tar pitches. This test is therefore valuable for purposes of identification.

Test 40. Conventional Method. The tar or pitch is first subjected to distillation in accordance with the retort method (Test 16*b*), the offtake and condensing tube being kept warm to prevent the accumulation of any solid distillate. The distillate passing over between 270 and 355° C. is caught separately and examined for anthracene in the following manner. The fraction is heated until it is thoroughly fluid to secure a uniform sample, and 5 g. weighed out, while hot. After cooling, 10 ml. of absolute ethyl alcohol are added, the solids allowed to crystallize and the liquid decanted. One to 2 g. of solid substances containing the anthracene are dried on a water-bath, transferred to a 500-ml. flask connected with a return condenser, 45 ml. of glacial acetic acid added, and the contents boiled for two hours. The following mixture is then added drop by drop through a separatory funnel, viz.: 15 g. of anhydrous chromic acid dissolved in 10 ml. of glacial acetic acid, and 10 ml. of water. The boiling is continued for another two hours, the flask cooled, and 400 ml. cold water added. This treatment oxidizes the anthracene to anthraquinone, which on cooling separates as a solid mass. This is filtered, washed with hot water, then with a hot 1 per cent solution of caustic soda and again with hot water. The residue of anthraquinone is then dried and its weight multiplied by

0.856 to obtain the corresponding weight of anthracene. From 0.25 to 0.75 per cent of anthracene is found in coal tars, and a correspondingly larger percentage in coal-tar pitches.

A color reaction for establishing the presence of anthracene consists in boiling for ½ to 1 hour the crystals of anthraquinone (1 part) with zinc dust (2 parts) and 50 per cent NaOH solution (30 parts), whereupon an intense red colored solution is obtained, which on filtering in contact with air becomes decolorized.

LIEBERMANN-STORCH REACTION

Test 41. Colorimetric Method. This is a rapid qualitative test for detecting the presence of rosin,[230] rosin oil, or cholesterol. One gram of the substance is dissolved in 3 ml. of acetic anhydride at a gentle heat, cooled and the clear liquid decanted into a porcelain crucible. Add 1 or 2 drops of sulfuric acid sp. gr. 1.53 (containing 62.53 per cent of pure sulfuric acid, prepared by diluting 34.7 ml. of concentrated sulfuric acid with 35.7 ml. of water). Rosin and rosin oil will produce a fugitive violet coloration turning to a brown, whereas cholesterol will produce a fugitive rose color turning rapidly to a dark green. If rosin or rosin oil is present in conjunction with cholesterol, the test becomes valueless.

Fossil resins (copals, etc.) also fatty-acid pitches give a permanent brown color and do not interfere with the foregoing test. Linseed, cotton-seed, china-wood and corn oils give a permanent greenish-brown coloration, whereas palm oil, bone tar, and crude olein give a permanent brownish-yellow coloration.

Highly heated rosin and rosin esters do not produce the characteristic color. The presence of rosin may be established with certainty by separating the rosin acids as follows: dissolve 5 g. of the substance in 150 ml. ether, filter, and shake the filtrate twice with 250-ml. portions of 1 per cent aqueous ammonium carbonate solution and then with 3 portions of 200 ml. each of 1 per cent caustic soda solution. Acidify the extract with hydrochloric acid, and extract the liberated resin acids with ether. Evaporate to dryness and dissolve the residue in 10 parts by weight of petroleum naphtha. Rosin is present if the violet color is produced when tested as above.

CHAPTER XXXIII

EXAMINATION OF BITUMINOUS SUBSTANCES COMBINED WITH DISCRETE AGGREGATES

Products falling into this class include native and artificial mixtures of bituminous matter with discrete aggregates, viz.: bituminous macadam pavements, bituminous concrete pavements, sheet asphalt pavements, asphalt block pavements, asphalt mastic floorings, bituminous expansion joints (containing mineral matter but not felt), pipe-sealing compounds, molding compositions and products used for electrical insulation.

(A) PHYSICAL TESTS OF FINISHED PRODUCT

(1) Paving Compositions, Asphalt Mastic, Bituminous Grouts, Pipe-Sealing Compounds, etc.[1]

Test 42. Specific Gravity. This is determined as described in Test 7, except that a larger specimen shall be taken, preferably cut as a cross-section of the completed material, and without the application of heat. The results express the "apparent specific gravity" of the composition, including any voids that may be present.

Test 43. Voids.[2] This test is adapted for testing compressed paving mixtures and supplements Test 7*f*, which may also be used for the purpose. The voids may be ascertained if the specific gravity and the per cent by weight of each constituent are known, as well as the apparent specific gravity of the mixture. Thus, if w_1, w_2 and w_3 represent the percentages by weight of sand, filler and asphalt, respectively, in a given mixture; and g_1, g_2 and g_3 their specific gravities, then the theoretically maximum density (D) of the composition (if entirely free from voids) may be calculated from the following formula:

$$D = \frac{100 g_1 g_2 g_3}{w_1 + w_2 + w_3}$$

The apparent specific gravity (d) of a test specimen having been ascertained, as in Test 42, the per cent of voids (v) may be calculated as follows:

$$v = \frac{100(D - d)}{D}$$

If w_0 = per cent by weight of total aggregate mixture
and g_0 = specific gravity of total aggregate mixture

Then $$v = 100 - d\left(\frac{w_0}{g_0} - \frac{w_3}{g_3}\right)$$

The per cent of voids (v_m) in the mineral aggregate of a compressed paving mixture (excluding the asphalt itself) is calculated from the expression:

$$g_m = d(1 - w_3)$$

Where g_m equals the apparent specific gravity of the mineral aggregate. The maximum theoretical density of the mineral aggregate (D_1) may be calculated from:

$$D_1 = \frac{w_1 + w_2}{\frac{w_1}{g_1} + \frac{w_2}{g_2}}$$

and $$v_m = \frac{D_1 - g_m}{D_1}$$

Test 44. Resistance to Moisture. Various methods have been suggested from time to time for ascertaining the water absorption of paving materials.[3] It is recognized that all pavements absorb more or less moisture, but no standard method has been proposed for this purpose. Clifford Richardson suggests the use of cylinders of the same dimensions as used for the impact test, namely 1.25 in. in diameter, by 1 in. high, having the greatest possible density, which in the case of surface mixtures for sheet asphalt pavements, will weigh about 50 g. They are immersed in water for three months, and the gain in weight noted at various intermediate periods.

Test 45. Effect of Water on Adhesion. The following method[4] has been proposed to determine the adhesion of asphalts to various types of mineral aggregate when subjected to the influence of water. The paving mixture is warmed until soft and then broken up with a

spoon or spatula while cooling. When cool, 50 g. are placed in a 250-ml. pyrex flask to which 175 ml. of water at 140° F. are added. The flask is then corked and rotated for 1 hour at the rate of 39 to 40 revolutions per minute while immersed in a water-bath maintained at 140° F. The flask is then removed and the sample examined to ascertain the extent to which the asphalt has been washed off the mineral aggregate, which ordinarily may be easily observed. In cases where this is not clearly visible, uncovered grains may be collected at the bottom of the flask, by rotating it a few times by hand, whereupon all the uncovered grains will congregate together.

The following fillers were found to have a greater affinity for water than for asphalt, viz.: silica (impure), infusorial earth, clay, iron oxide, aluminium oxide (bauxite), impure limestone, calcined magnesia, gypsum, barytes, lithopone, granite containing feldspar, and certain kinds of slag. The following fillers have a greater affinity for asphalt than for water, viz.: pure silica (e.g., crushed Ottawa sand), pure limestone, hydrated lime, powdered limestone rock-asphalt, and calcined coal-mine waste. Even with pure limestone, Bermudez asphalt adheres much better than do petroleum asphalts. Natural limestone rock-asphalts from Texas, Sicily, Germany, France and Alabama adhere very much better than do petroleum asphalts.

Test 46. Hardness. The following test has been proposed[5] for this purpose. Test pieces are prepared in the form of cylinders 2 in. in diameter and 2 in. high, either by cutting from the solid mixture by means of a carborundum saw, or by molding the material, warmed to 130–140° F. under sufficient pressure (about 12,000 lb. per sq. in.), preferably in a mold with a double plunger acting on top and bottom of the specimen, so that after compression it will have the same gravity as the original mixture. After molding, the test pieces are allowed to age at room temperature for 7 days. The hardness is defined as the depth expressed in hundredths of a cm., to which a steel rod of 6.35 mm. diameter, with the end cut off square, will penetrate the test piece in 60 seconds at 77° F., under a load of 100 kg. per sq. cm. The testing machine is illustrated in Fig. 286. A vertical rod has attached to its lower end a smaller steel rod 6.35 mm. in diameter. The horizontal beam is pivoted near its center, and counter-balanced by the weight shown

at the left of the illustration. The test specimen is maintained in a water-bath at 77° F. The load is applied by placing the right-hand weight at a predetermined position on the beam. The distance penetrated is indicated by a vernier gear attachment, which records the depth in hundredths of a cm. The right-hand weight may be fixed at different positions to give different loads, and the temperature may be varied, as desired. An average of 6 separate readings is taken, being careful to keep away from the edge of the specimen.

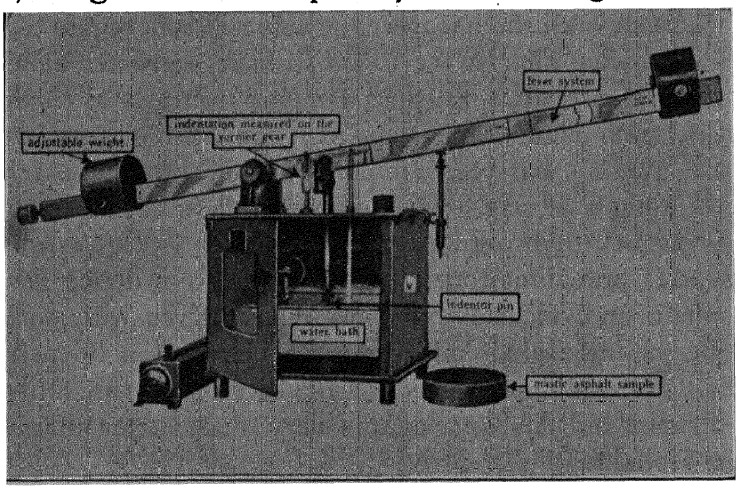


FIG. 286.—Hardness or Resistance to Indentation Carried Out Directly on the Finished Mastic Asphalt.

It consists essentially of an electrically heated oven the interval temperature of which can be controlled accurately.

The vertical movement of the indentor pin may be measured accurately by a vernier gear. A known load can be applied to this indentor pin by means of a lever system. The load is applied for a period of one minute.

A similar procedure consists in measuring the distance in which a steel ball will penetrate into the substance under a given loading and temperature.[6]

Test 47. Resistance to Displacement. This test has been proposed[7] for examining fine-aggregate paving mixtures (e.g., the wearing-course of sheet-asphalt pavements) and has also been adapted for testing coarse-aggregate paving mixtures.[8] The procedure when testing fine-aggregate mixtures consists in weighing the dry sand and filler to 0.1 g.; heating to 350° F. and mixing thoroughly. The melted asphaltic binder heated to 350° F. is then

weighed to 0.1 g. and thoroughly mixed with the aggregate by means of a spatula. Approximately 100 g. of the completed mixture are weighed into a cylindrical forming mold 2 in. in diameter and 4 in. high. The mold and contents are heated to 250–260° F. as determined by the thermometer used for stirring the mixture. Remove and insert a cylindrical plunger into the mold, and then tamp by first giving 60 blows with a tamper weighing 500 g., fol-

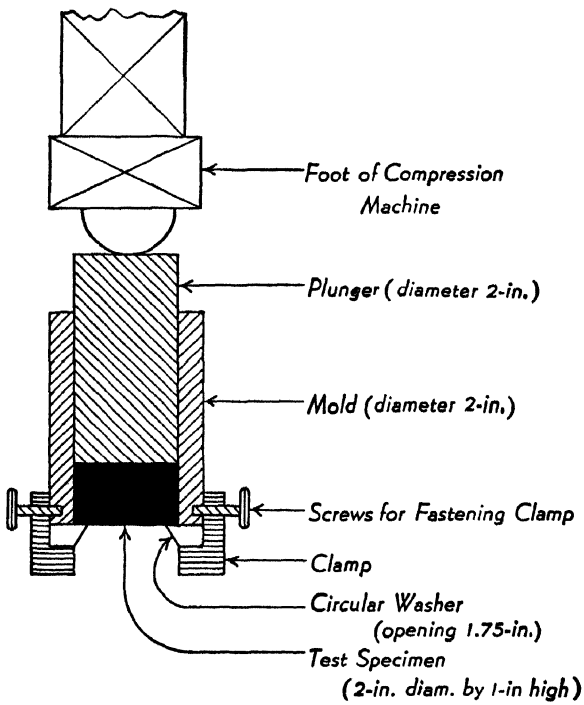


FIG. 287.—Apparatus for Testing Resistance to Displacement.

lowed by 15–20 blows with a tamper weighing 1400 g. The mold and contents are placed in a compression machine to which is applied a load of 3000 lb. per sq. in. and the briquette allowed to cool under pressure. The compressed briquette should measure 2 in. in diameter and approximately 1 in. in height. Let stand at room temperature over night and then maintain in a water-bath at 140° F. for one hour before testing. Two such briquettes should be prepared, each specimen being tested by placing it top end down in the testing mold and inserting the plunger as illustrated in Fig. 287. A circular washer having a circular opening 1.75 in. in diameter is

clamped on the lower end of the mold and the load then applied at a rate which will cause the testing head to be depressed 1 in. in 25 seconds. As the pressure increases, the briquette will be forced through the orifice of the circular washer. The reading is observed on a spring dynamometer and the maximum is recorded. The average of two specimens is taken as a measure of the "stability" of the mixture. This test indicates the resistance of the pavement to displacement and may be used to proportion mixtures of sand, filler and asphaltic cement giving optimum results.

For testing coarse-aggregate paving mixtures [9] a larger mold is recommended in conjunction with a testing ring ⅜ in. thick, having a minimum orifice 5¾ in. in diameter for a depth of ⅛ in., after which it tapers out to a diameter of 6 in. at the bottom of the ring.

Samples of the finished mixture taken from the testing plant are warmed to 250–260° F. and formed into briquettes as described above. Finished pavements may be tested by cutting out cores 2 in. in diameter and approximately 1¼ in. high, which are then placed directly in the mold and tested at 140° F.

Test 48. Extrusion of Binder under Pressure. A rapid test has been proposed [10] for ascertaining by colorimetric means any excess of bituminous binder present in paving mixtures by means of a so-called "compression stain print." A briquette of the paving mixture, of cylindrical form, 1 to 2 in. high having a cross-sectional area of 3 sq. in., is prepared as in Test 47 and subjected to a pressure of 6000 lb. for 12 minutes at a temperature of 300–375° F., one surface being placed in contact with a sheet of kraft paper. The stain produced on the paper may be observed as an indication of the excess or deficiency of the bituminous binder present in the mixture. Thus the degree of discoloration or stain is proportionate to the relative amount of binder present, and an experienced observer will quickly learn to judge by eye whether there is an excess or deficiency of binder, and approximately the amount of such excess or deficiency.

Test 49. Resistance to Impact. This test was originally devised by L. W. Page for testing the toughness of rock for road building,[11] having since been adapted by Clifford Richardson for testing bituminous aggregates.[12] The bituminous mixture is heated to the lowest possible temperature that will permit it being manipu-

lated, and formed by compression into a cylinder 25 mm. high by 24–25 mm. in diameter, the ends of which shall be plane surfaces at right angles to its axis. The hot bituminous mixture is compressed in a hollow cylindrical steel mold, 24–25 mm. in diameter by 50 mm. long, having an accurately fitting steel plunger. The mold is loosely filled with the hot bituminous mixture and compressed with the plunger by sharp blows of a heavy hammer from the top and bottom respectively, until it is thoroughly compacted. The cylinder of bituminous material is then knocked from the mold and sawed off or ground down until it measures exactly 25 mm. high. The density of the specimen should be noted and reported. It shall be maintained in water at 77° F. for 48 hours, wiped dry, and tested in air at a temperature of 77° F. on any form of impact machine which will comply with the following essentials:

(*a*) A cast-iron anvil weighing not less than 50 kg. firmly fixed upon a solid foundation.

(*b*) A hammer weighing 2 kg. arranged to fall freely between suitable guides.

(*c*) A plunger of hardened steel weighing 1 kg. arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical, with a radius of exactly 1 cm.

(*d*) Means for raising the hammer and dropping it upon the plunger from any specified height from 1 to not less than 75 cm.

(*e*) Means for holding the cylindrical test-specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall, throughout the test, be tangent to the spherical end of the plunger at its lowest point.

The test shall consist of a 1-cm. fall of the hammer for the first blow; a 2-cm. fall for the second blow; and an increase of 1 cm. for each succeeding blow, until failure of the test specimen occurs. The number of blows required to shatter the test-piece is taken to represent the toughness, three such tests being averaged. Tests are performed at three temperatures, viz.: 32° F., 77° F. and 115° F.

Another method has been proposed for testing pavements,[18] in which specimens of full thickness are constructed as slabs 3 ft. square, upon a rolled cinder subgrade, and tested in place by allowing a 125-lb. iron ball to drop from a height of 6 in. at the rate of 30 blows per minute for the first 1000 blows, 7 in. for the next 200

blows, and an increase of 1 in. for each successive 200 blows until the first crack appears, which is regarded as the failure of the specimen. Fig. 288 shows the behavior of various types of pavements under this test. Concrete specimens were cured under moist sand and tested when twenty-eight days old.

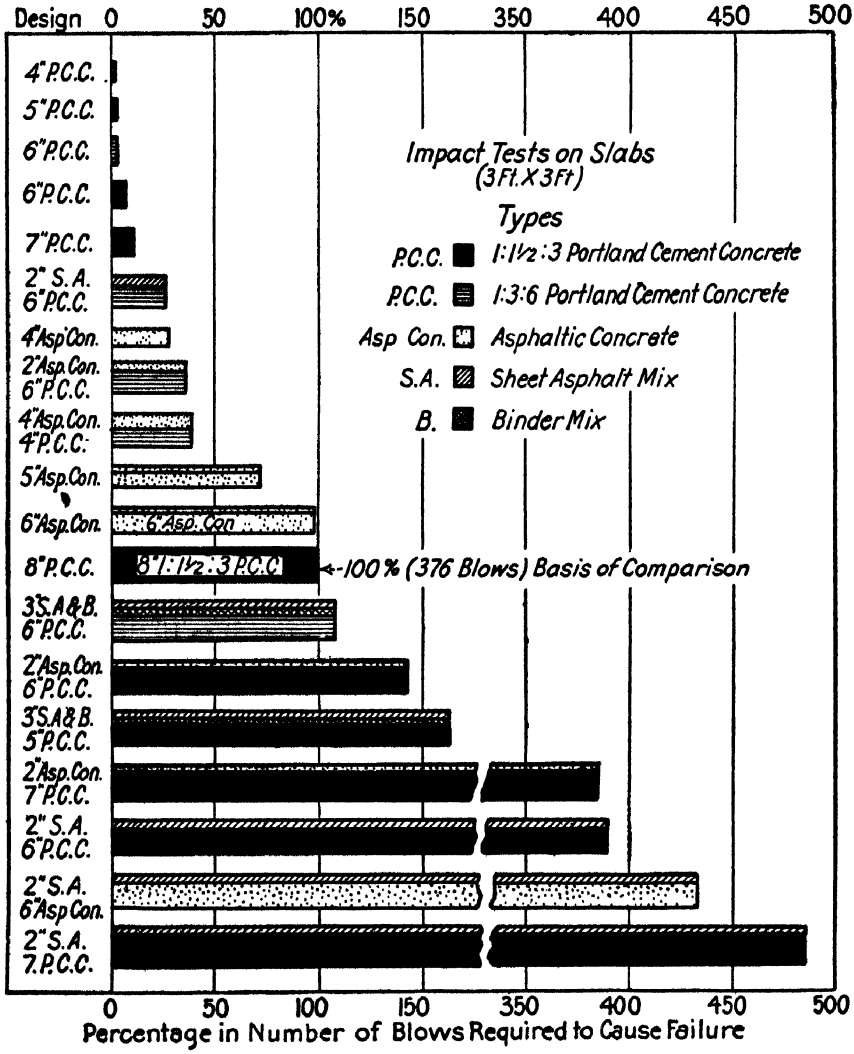


FIG. 288.—Resistance of Typical Pavements to Impact.

Test 50. Brittleness or Shatter Test.[14] This shall be performed as described in Chapter XXVII, under the heading "Bituminous Enamel for the Inside of Steel Ships," subheading "Brittleness or Shatter Test."

Test 51. Coefficient of Wear. This may be measured by a sandblast under carefully controlled conditions.[15] Sand of predetermined size is projected against an area 6 cm. in diameter, under a pressure of 3 atmospheres. For further details, the reader is referred to the article given in the reference.

Another test has been proposed[16] in which a section of the pavement is laid in a 14-ft. diameter circular track and subjected to the action of a metal roller, or to a pneumatic tired wheel under accurately controlled conditions.

(2) Molded Materials

Test 51a. Thickness. This test has been standardized as follows:[17] Two alternative procedures, Methods A and B, are described. In Method A the use of an adjusted ratchet micrometer and a definite manipulative procedure controls the pressure exerted on the specimen. In Method B a micrometer without ratchet is used and pressure on the specimen is controlled by stopping closure of the micrometer when resistance to movement of the specimen between the instrument faces is first observed.

Method B is used generally for routine measurements. Method A, which may also be used for routine measurements, is preferable in cases of dispute. The precisions of the two methods are as follows:

For unyielding materials, such as porcelain and laminated or molded phenolic materials, the maximum error of either method is of the order of 0.5 mil or less.

For compressible materials, such as paper, fabric or leather, the maximum error of Method A is of the order of 0.5 mil. By applying the proper technique, Method B will give an accuracy of the order of 0.5 mil for the usual thicknesses of resilient materials encountered in the trade, except that for materials which are very compressible, the error may be somewhat greater.

Method A

The instrument used for determining thickness by Method A shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with a vernier reading to 0.1 mil (0.0001 in.) and with a ratchet or similar mechanism for controlling meas-

ured pressure, and shall have anvil and spindle surfaces 0.250 in. ± 0.001 in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, wear of micrometer screw, micrometer screw error tolerances and ratchet pressure specified below. The micrometer shall be tested and calibrated periodically for conformity to these requirements.

Before starting measurements of thickness the micrometer shall be closed on the specimen at a location outside the area to be measured. The micrometer shall then be opened not more than 4 or 5 mils (0.004 or 0.005 in.) and then moved into the area selected for measurement. Using the ratchet, the micrometer surfaces shall be closed so slowly on the specimen that the mil scale divisions may be easily counted as they move past the reference mark, or at a rate of about 2 mils (0.002 in.) per second. The closing motion shall be continued at the same rate until the ratchet has clicked three times, and then the thickness shall be read by means of the vernier.

In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or 0.005 in.) more than the specimen thickness.

Method B

The instrument used for determining the thickness by Method B shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with vernier reading to 0.1 mil (0.0001 in.) and with anvil and spindle surfaces 0.250 in. ± 0.001 in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, and micrometer screw error tolerances specified below. The micrometer shall be tested and calibrated periodically for conformity to these requirements.

In the determination of thickness the micrometer specified shall be slowly closed on the specimen until contact is made, without appreciable distortion of the specimen. The criterion of contact is the initial development of frictional resistance to movement of the specimen between the micrometer surfaces. The thickness shall then be read by means of the vernier.

In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or 0.005 in.) more than the specimen thickness. Before and during instrument calibration and thickness measurements, the micrometer surfaces shall be maintained in a clean condition by lightly closing them on a clean sheet of bond paper and moving the paper between the surfaces. To minimize the danger of the presence of lint, the edge of the paper should not be pulled between the surfaces.

The manipulative procedure for calibrating controlled pressure instruments used in Method A, shall be in accordance with the following:

The micrometer shall be closed on the gage or calibrating device and then opened 4 or 5 mils (0.004 or 0.005 in.). Using the ratchet, the micrometer shall be again closed so slowly on the calibrating device that the mil scale divisions may be easily counted as they move past the reference mark, or at the rate of about 2 mils (0.002 in.) per second. The closing motion shall be continued at the same rate until the ratchet has clicked three times, when the reading shall be taken.

The manipulative procedure for calibrating instruments used in Method B shall be in accordance with the following: The micrometer shall be slowly closed on the gage or calibrating device until contact of the surfaces and gage is made. The criterion of contact is the initial development of frictional resistance to movement of the gage device between the micrometer surfaces.

The anvil and spindle surfaces of the micrometer shall be flat to within 0.00005 in. The flatness may be determined by use of an optical flat, as follows: After cleaning the surfaces of the flat and the micrometer the latter shall be closed on the flat as described above. When illuminated by diffused daylight, interference bands are formed between the surfaces of the flat and those of the micrometer. The location, shape and number of these bands indicate the deviation from flatness in increments of half the average of the wave lengths of white light, which is taken as 0.00001 in.

A *flat surface* forms straight, parallel and equidistant fringes.

A *grooved surface* forms straight parallel fringes at unequal

intervals. The estimated maximum displacement of any line from its normal position, where all lines would be equidistant, is a measure of deviation from flatness.

A *symmetrical concave or convex surface* forms concentric circular fringes, and their number is a measure of deviation from flatness.

An *unsymmetrical concave or convex surface* forms a series of curved fringes, cutting the periphery of the micrometer surface. The number of fringes cut by a straight line connecting the termini of any fringe is a measure of the deviation from flatness.

The anvil and spindle surfaces of the micrometer shall be parallel to each other to within 0.0001 in. when tested with a pair of screw-thread-pitch wires or with a pair of ¼-in. nominal diameter plug gages. The diameters of the screw-thread-pitch wires or the plug gages, accurate to 0.00002 in., shall differ by an amount approximately equal to the axial movement of the spindle when rotated through 180 deg. (12.5 mils). The micrometer shall be closed on the wires or on the plug gages according to the procedure described above. Observations, made with either wire or with either plug gage placed at any location between the surfaces, shall show differences of less than 0.0001 in. The position of the anvil shall be such that a zero reading is obtained when the micrometer is closed on the anvil as described above. Ten trials shall give ten readings of zero.

The device for compensating for wear of the micrometer screw shall be adjusted so that the spindle has no perceptible lateral or longitudinal looseness and yet may be rotated with a torque load of not more than ¼ in.-oz.

The micrometer screw error, after zero adjustment is made, shall be checked at 2, 5, and 10 mils, and at intervals of 100 mils over the remaining graduated scale. For checks up to and including a thickness of 10 mils, selected gage blades the thicknesses of which are known to ± 0.00002 in. shall be used. Checks at values greater than 10 mils shall be made with standard gage blocks. At each value checked, ten readings shall not differ from the thickness of the gage used by more than 0.1 mil (0.0001 in.). Manipulation of the instrument in these checks shall be in accordance with the above. The ratchet shall be so adjusted that a pressure of not more

than 27 nor less than 23 lb. per sq. in. is developed when the spindle surface is contacted with a polished steel surface as described.

Test 52. Resistance to Moisture. The following method has been standardized:[18] The test specimen shall be molded in the form of a disk 4 in. (10.16 cm.) in diameter, which should be 1/8 in. (3.18 mm.) in thickness for hot-molded materials, and 1/4 in. (6.35 mm.) in thickness for cold-molded materials.

NOTE. To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds.....± 5 per cent
For cold-molded compounds.....± 10 per cent

Three specimens shall be tested. Each of the three shall be weighed separately in the as-received condition. If the material softens readily at moderate temperatures, the specimen may be placed in a desiccator for twenty-four hours, or in an oven at a temperature of 50° C. (122° F.), permissible variation ± 5° C., for twenty-four hours. For materials which do not soften readily, the specimen shall be placed in an oven heated to 100° C. (212° F.), permissible variation ± 5° C., for twenty-four hours. After drying, the pieces shall be cooled in a desiccator and weighed again at normal room temperature. The specimens shall be placed in water, wholly immersed, for forty-eight hours at a temperature of 25° C. ± 2° C. They shall then be removed from the water at the end of forty-eight hours, all surface water wiped off with a dry cloth, and the specimens weighed immediately.

The report shall include the following:

(*a*) The original weight of each specimen;
(*b*) The dry weight of each specimen;
(*c*) The weight of each specimen after immersion for forty-eight hours;
(*d*) The percentage of water contained in each test specimen as received, and the percentage of moisture absorbed during the forty-eight hours, taking the dry weight as 100 per cent.

All weights shall be given in grams.

The following alternate procedure has been proposed[19] for testing sheet and plate materials intended for electrical insulation:

(*A*) *Rate of Absorption.* The test specimen shall be 3 by 1 in.

by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. Sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches to the nearest 0.001 in. shall be measured on the original specimen at the center.

To determine the rate of absorption, two specimens shall be tested individually as follows: The specimen shall be weighed, dried in an oven for 1 hour at from 105 to 110° C., cooled in a desiccator and immediately re-weighed. The difference between the original weight and the dry weight shall be recorded as loss in weight on drying. The percentage loss in weight shall be calculated on the original weight.

The dried specimen shall then be immersed in distilled water maintained at a temperature of 25° C. ± 2° C. (77° F. ±3.6° F.) throughout the test. At the end of 2 hours the specimen shall be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed immediately and then replaced in the water. If the specimen is 1/16 in. in thickness or less it shall be weighed in a weighing bottle. The difference between the weight of the wet specimen and that of the dry specimen shall be recorded as the weight of water absorbed in 2 hours. The percentage of water absorbed shall be calculated on the weight of the dry specimen.

After 24 hours' immersion the specimen shall again be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed. The difference between the weight of the specimen after 24 hours' immersion and the weight of the dry specimen shall be recorded as the water absorbed in 24 hours. The percentage of water absorbed shall be calculated on the weight of the dry specimen. If the individual 24-hour absorptions differ by more than 10 per cent from the average absorption of the two specimens the test shall be repeated.

The report shall include the values for each of the two specimens and the average values as follows:

(*a*) The thickness of the specimen before the test, to the nearest 0.001 in.;

(*b*) The percentage loss in weight on drying calculated on the original weight;

(*c*) The percentage of water absorbed in 2 hours and in 24 hours calculated on the dry weight.

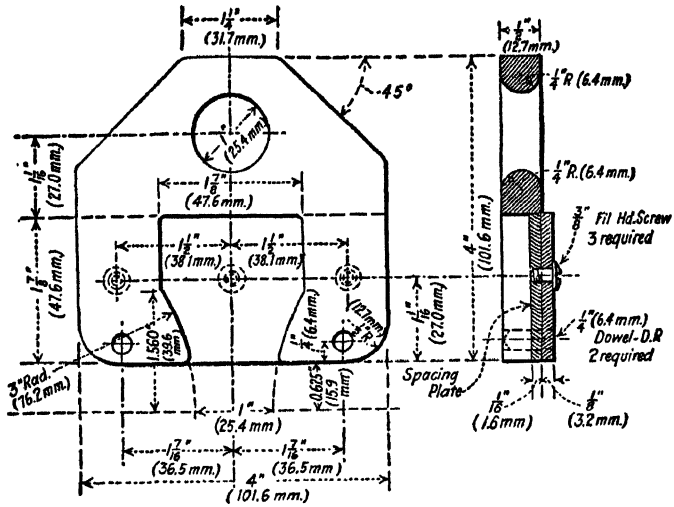
(*B*) *Total Absorption at Saturation.* The test specimen shall be 3 by 1 in. by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. Sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches measured to the nearest 0.001 in. shall be measured on the original specimen at the center and on the reduced specimen in case a reduced specimen is used (see following paragraph).

When the sample for determining total absorption is so thick that too long a time is required for saturation using the full thickness, the thickness may be reduced as follows: The sample shall be machined to a thickness of about $\frac{5}{64}$ in., both surfaces parallel to the laminations being machined off. This may readily be done with a smooth-cut circular saw. It shall then be sanded to $\frac{1}{16}$ in. in thickness, finishing with No. 0 or finer sandpaper. While the sanded surfaces of the sample should be approximately parallel with the laminations, the thickness may vary $\pm$ 0.010 in., since the object is to provide comparable specimens which will reach saturation in a reasonable time. The machined and sanded sample shall then be cut into specimens 3 by 1 in. and the edges sanded.

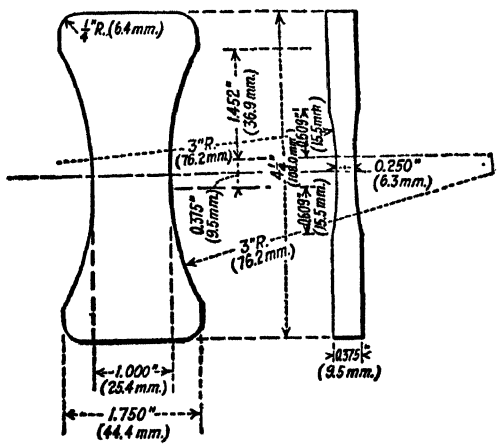
To determine the total water absorption at saturation, two specimens shall be tested individually as follows: The specimen shall be weighed, dried in an oven for 1 hour at from 105 to 110° C., cooled in a desiccator and immediately reweighed. The difference between the original weight and the dry weight shall be recorded as loss of weight on drying. The percentage loss in weight shall be calculated on the original weight.

The dried specimen shall then be immersed in distilled water at a temperature of from 20 to 30° C. At the end of 24 hours the specimen shall be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed immediately and then replaced in the water. The weighings shall be repeated every day

for the first week and every week thereafter until the increase in weight, as shown by three consecutive weighings, averages less than



(a) Specimen holder.



(b) Tension test specimen.

Courtesy A.S.T.M.

FIG. 289.—Tension Test Specimen and Holder.

NOTE.—To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds	± 5 per cent
For cold-molded compounds	± 10 per cent

1 per cent of the total increase in weight, when the specimen shall be considered to be saturated.

The difference between the saturated weight and that of the dry weight shall be considered as the water absorbed at saturation. The

percentage of water absorbed shall be calculated on the dry weight of the specimen.

This report shall include the values for each of the two specimens as follows:

(*a*) The original thickness of material, measured to the nearest 0.001 in.;

(*b*) If a reduced specimen is used, the thickness measured to the nearest 0.001 in.;

(*c*) The percentage loss in weight on drying, calculated on the original weight;

(*d*) The percentage of water absorbed at saturation, calculated on the dry weight.

Test 53. Tensile Strength. This has been standardized as follows:[18]

Any standard testing machine may be used provided it is accurate to within 1 per cent of the lowest load to be applied. Special specimen holders of steel, as shown by Fig. 289(a), shall be used. The specimen holders shall be attached to the heads of the machine by links held so that the pull is central, to avoid any transverse strain. For hot-molded materials and for plastic cold-molded materials, the test specimen shall be molded to the form and dimensions shown in Fig. 289(b).

All specimens shall be conditioned before testing by drying in an oven at 50° C. ± 5° C. for 48 hr., followed by cooling to room temperature in a desiccator. Five specimens shall be tested at room temperature. The speed of the head of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. (1.27 mm.) per minute with the machine running idle. All tests shall be made at normal room temperature of about 20° C. (68° F.).

The report shall include the following:

(*a*) The breaking load of each specimen in pounds or kilograms;

(*b*) The thickness and width of each specimen in inches or millimeters as measured by a micrometer at the center of the specimen, that is, the point of minimum section;

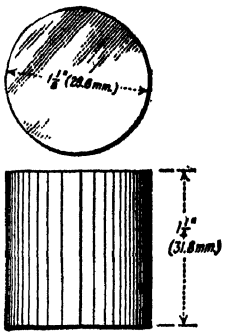
(*c*) The ultimate tensile strength in pounds per square inch or in kilograms per square millimeter of each specimen, calculated

from the minimum area measured at the center of the test specimen before the load is applied;

(*d*) The character of the material tested, with description of the fracture and its location with respect to the middle point of the specimen.

(*e*) The speed in inches or millimeters per minute at which the head of the testing machine traveled during the test.

Test 53a. Compressive Strength. This method has similarly been standardized as follows:[18]



Courtesy A.S.T.M.

FIG. 290.—Compression Test Specimen.

Any standard testing machine may be used provided it is accurate to within 1 per cent of the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top, and the metal bearing plates shall be directly in contact with the ends of the test specimen.

The test specimen shall be molded in the form of a cylinder having a height of 1.25 in. (31.8 mm.) and a diameter of 1.125 in. (28.6 mm.) (Fig. 290). The ends shall be flat and perpendicular to the axis, being ground to this condition if necessary.

NOTE 1. To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds	± 5 per cent
For cold-molded compounds	± 10 per cent

NOTE 2. Due to the size and shape of this specimen, it is sometimes difficult to completely cure it within the relatively short time which would ordinarily be used and with the temperatures ordinarily used. This is particularly true when hand molds, that is, molds heated by conduction from the press platens, are used. It is, therefore, recommended that special precautions be taken to insure thoroughly cured parts, experiments indicating that 20 to 30 minutes may be necessary in some cases with hand molds.

All specimens shall be conditioned before testing by drying in an oven at 50° C. ± 5° C. (122° F. ± 9° F.) for 48 hr., followed by cooling to room temperature in a desiccator. Five specimens shall be crushed at room temperature.

Additional tests shall be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each tem-

perature selected. The specimen shall be kept at that temperature a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

NOTE 1. A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically-heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least 30 minutes before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ± 10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial-immersion thermometer conforming to the Standard Specifications, so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

NOTE 2. The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength determination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least 30 minutes before testing. An average curve should be drawn through the plotted data.

The load shall be applied on the ends of the specimen and the cross-head speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle. For the best results use the slowest possible speed.

The report shall include the following:

(*a*) The dimensions of each specimen in inches or in millimeters;

(*b*) The load on each specimen in pounds or kilograms at the first sign of failure;

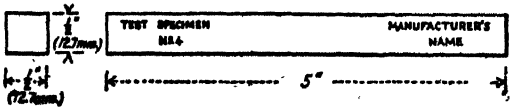
(*c*) The ultimate compressive strength in pounds per square inch or kilograms per square millimeter of each specimen, calculated from the measured area of each specimen before the load is applied;

(*d*) The general character of the material tested, with description of how it acts under the applied load;

(*e*) The speed in inches or millimeters per minute at which the head traveled during the test. The time required for testing each specimen should be recorded.

Test 54. Flexural Strength. This test has been standardized as follows:[18]

Any standard testing machine may be used provided it is accurate to within 1 per cent for the lowest load for which it is used. The distance between points of support shall be 4 in. (102 mm.). The supports shall have the contact edges rounded to a radius of 1/8 in. (3.18 mm.). The load shall be applied parallel to and in the same direction that the molding pressure was applied, midway between the supports by a pressure piece, the bearing edge of which shall be rounded to a radius of 1/8 in. (3.18 mm.). The test specimen shall be molded to the dimensions shown in Fig. 291.



Courtesy A.S.T.M.

FIG. 291.—Test-Specimen for Flexural Strength.

All specimens shall be conditioned before testing by drying in an oven at 50° C. ± 5° C. (122° F. ± 9° F.) for 48 hr., followed by cooling to room temperature in a desiccator. Five specimens shall be tested at room temperature.

The cross-head speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle. Measurements of the deflection may be taken for very elastic materials.

The report shall include the following:

(*a*) The thickness and width of each specimen as measured by a micrometer in inches or millimeters;

(*b*) The load on each specimen in pounds or in kilograms at the first sign of failure;

(*c*) The maximum fiber stress in pounds per square inch or in kilograms per square millimeter calculated from the formula:

$$S = \frac{3Pl}{2bd^2}$$

in which S = maximum fiber stress, P = load applied, l = distance between the supports, b = width of specimen, and d = depth of specimen;

(*d*) The rate at which the load was applied;

(*e*) The maximum deflection at the center in inches or in millimeters.

Test 55. Distortion under Heat. The following method has been standardized:[18]

A special apparatus shall be used for this test as shown in Fig 292. The specimen is supported on steel supports, 4 in. (102 mm.) apart with the load applied on top of the specimen vertically and midway between the supports, the same as for the transverse test. The machine shall be arranged to apply a load of 5 lb. (2.5

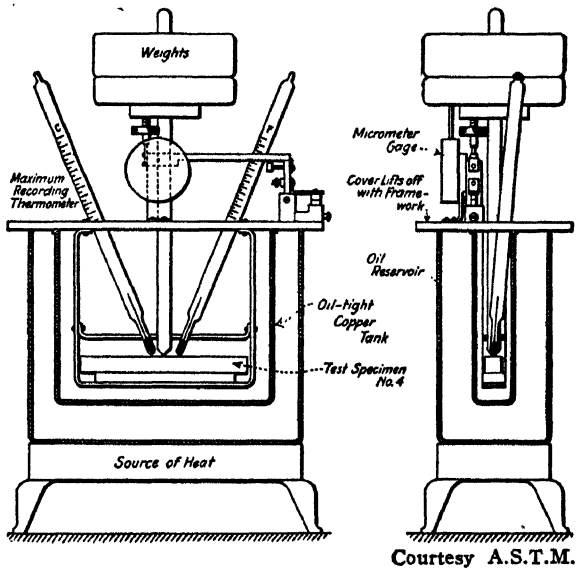


FIG. 292.—Machine for Distortion under Heat.

kg.). The specimen shall be placed in an air-bath surrounded by an oil-bath which is so arranged that its temperature may be raised gradually. The machine shall be so arranged that the deflection of the specimen at its center between the supports can be measured on a scale in mils or millimeters and shall be equipped with a thermometer so that the temperature of the specimen can be recorded at any time. The machine may be arranged automatically to shut off the heat and sound an alarm as soon as the required deflection is reached.

The same test specimens shall be used for this test as required for the transverse strength test, molded to the dimensions shown in Fig. 291. Three test specimens shall be tested in the condition in which they are received, starting at normal room temperature of about 20° C. (68° F.) and increasing the temperature gradually

at a rate not exceeding approximately 1° C. every two minutes. The distortion point shall be considered the temperature at which the specimen has deflected 10 mils (0.254 mm.) at the center between the supports.

The report shall include the following:

(*a*) The breadth and depth of each specimen measured at the center with a micrometer in inches or in millimeters;

(*b*) The distortion point in degrees Centigrade or in degrees Fahrenheit;

(*c*) The length of time in minutes required for each specimen to deflect 10 mils (0.254 mm.);

(*d*) Any peculiar characteristics of the material as noted either during the test or after the specimen is removed from the machine.

Two curves may be plotted, one on a chart showing the minutes horizontally, the deflection in mils shown vertically to the left, and the temperature in degrees shown vertically to the right. One curve represents the deflection in mils at given time intervals, and the other represents the temperature at given time intervals.

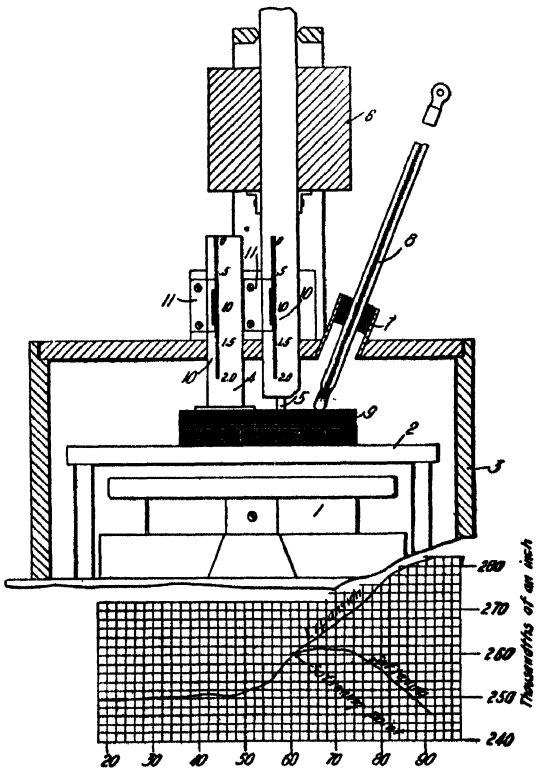


FIG. 293.—Apparatus for Determining the Softening-point of Bituminized Aggregates.

Test 56. Softening-Point. An ingenious apparatus for determining the softening-point of molded insulating materials, which is likewise adapted to testing pavements, asphalt mastic floorings, expansion joints and pipe-sealing compounds, has been devised by H. R. Edgecomb [20] as illustrated in Fig. 293. The underlying principle consists in comparing the expansion with the tendency to soften as the tem-

perature increases. The apparatus consists of an electrical heater 1, a plate or slab 2 above the heater, a hood 3 for retaining the heat, a rod 4 having a relatively large lower face resting loosely on the specimen, a rod 5 having a relatively small lower face (0.01 sq. in. in area) actuated by a weight 6 of either 2.5 or 5.0 kg., and an opening 7 for the thermometer 8. The rods and thermometer rest upon the insulating material 9 to be tested, and each of the rods 4 and 5 is provided with a scale 10 operating in conjunction with stationary vernier scales 11, for recording the movement.

It is important that the sample 9 be provided with two plane faces, also that the temperature is increased at the uniform speed of 1° F. per minute. The positions of the rods 4 and 5 are noted at periodic intervals, and two curves plotted with the temperature abscissas and the movement of the rods respectively in thousandths of an inch as ordinates. These curves will be identical as the material expands throughout a certain range in temperature, but when it begins to soften, rod 5 will change its direction of travel, and instead of moving upward will embed itself in the sample. The point at which the two curves diverge represents the softening-point of the material. This is shown at 60° F. in the chart illustrated in Fig. 293.

Test 57. Resistance to Impact. Special methods have been devised for this purpose,[21] the details of which are as follows:

These methods are intended to determine the relative toughness of electrical insulating materials as indicated by the energy used in breaking a standard test specimen in one blow.

This type of impact test is made by machines of the pendulum type of which there are two kinds, the Charpy and the Izod, which differ only in the method of holding and striking the specimen. In the Charpy test the specimen is held as a simple beam (usually horizontal) and is broken by a blow delivered midway between the supports. In the Izod test the specimen is held as a cantilever beam (usually vertical) and is broken by a blow delivered at a fixed distance from the edge of the specimen clamp. The Charpy test may be made on either a plain or a notched specimen as required by the characteristics of the material tested. The Izod test requires a notched specimen in all cases.

Simple Beam Method (Charpy Test). The machine used in the simple beam method shall be of the pendulum type as shown in Fig. 294, of rigid construction and accurate to within 0.01 ft.-lb. for values of impact strength less than 1 ft.-lb. and to within 1 per

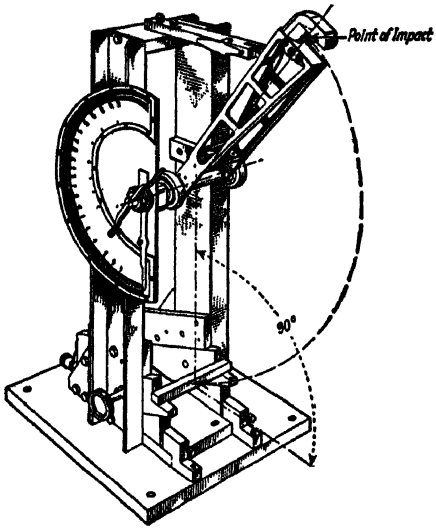
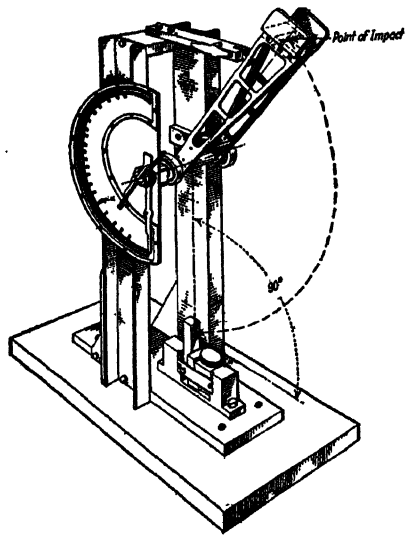


FIG. 294.—Simple Beam (Charpy) Impact Machine.



Courtesy A.S.T.M.

FIG. 295.—Cantilever Beam (Izod) Impact Machine.

cent for higher values of impact strength. Accurate correction shall be made for friction and windage losses.

The dimensions of the machine shall be such that the center of percussion of the striker (see Note) is at the point of impact, that is, the center of the striking edge.

NOTE. The distance from the axis of support to the center of percussion, l, may be determined experimentally from the period of oscillation of the pendulum through a small angle by means of the expression $l = 0.81\ P^2$, where P is the time in seconds of a complete swing (to and fro).

The pendulum shall be released from such a position that the linear velocity of the center of the striking edge (center of percussion) at the instant of impact shall be approximately 11 ft. per second, which is that corresponding to an initial elevation of this point of 2 ft.

The striking edge of the pendulum shall be tapered to have an included angle of 45 deg. and shall be rounded to a radius of 0.125

in. (3.175 mm.). It shall be so aligned that in the case of rectangular specimens it will make contact across the full width of the specimen.

The test specimen shall be supported against two rigid blocks in such a position that its center of gravity shall lie on a tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The edges of the blocks shall be rounded to a radius of 0.125-(3.175 mm.) and the points of support shall be 4 in. (10.16 cm.) apart.

Means shall be provided for determining the impact strength. The impact strength shall be taken as the energy absorbed in breaking the specimen, and is equal to the difference between the energy in the pendulum blow and the energy remaining in the pendulum after breaking the specimen.

Cantilever Beam Method (Izod Test). The machine used for the cantilever beam method shall be of the pendulum type as shown in Fig. 295, of rigid construction and accurate to within 0.01 ft.-lb. for values of impact strength less than 1 ft.-lb. and to within 1 per cent for higher values of impact strength. Accurate correction shall be made for friction and windage losses. The dimensions of the machine shall be such that the center of percussion of the striker is at the striking edge.

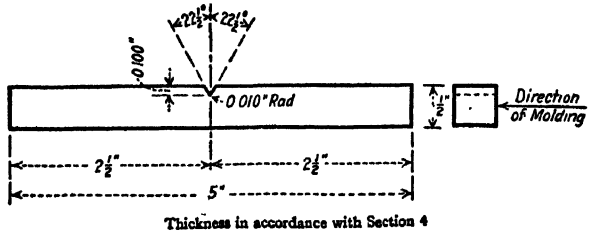
The pendulum shall be released from such a position that the linear velocity of the striking edge at the instant of impact shall be approximately 11 ft. per second, which is that corresponding to an initial elevation of the striking edge of 2 ft.

The striking edge of the pendulum shall be an unrounded edge formed, in the position of impact, by the intersection of a plane inclined away from the specimen at an angle of 5 deg. from the vertical with another plane perpendicular to the specimen, and shall be so aligned as to make contact across the full width of the specimen. In the striking position the striking edge shall be 0.866 in. from the edge of the specimen clamp.

Means shall be provided for clamping the specimen rigidly in position with the edges of the supporting surfaces at 90-deg. angles. Means shall be provided for determining the impact strength. The impact strength shall be taken as the energy absorbed in breaking the specimen, and is equal to the difference between the energy in

the pendulum blow and the energy remaining in the pendulum after breaking the specimen.

Charpy Test Specimen. For insulating materials other than ceramic materials the specimen shall conform to the dimensions shown in Fig. 296. The notch, when used, may be milled, filed, or molded provided the bottom of the resulting notch is smooth, straight and free from scratches. For molded material (see Note 1) the thickness of the molded specimen, that is, the dimension parallel to the application of the molding pressure and lengthwise of the notch, may be any thickness of ½ in. or less agreed upon as representative of the cross-section in which the particular material



Courtesy A.S.T.M.

FIG. 296.—Simple Beam (Charpy) Impact Test Specimen.

is to be used. For sheet material the thickness shall be the thickness of the sheet except that it shall not exceed ½ in. Sheet material, when thicker than ½ in. may be tested either edgewise or flatwise, as specified (see Note 2), and when tested flatwise, the notch shall be made in the original surface. When the individual specimen is less than ½ in. in thickness, the test piece shall be a composite specimen consisting of a number of individual thin specimens aggregating as nearly as possible ½ in. in thickness. They shall all be accurately aligned with each other and shall be tested edgewise. Specimens of molded insulating material shall be conditioned before testing by drying in an oven at 50° C. ± 5° C. (122° F. ± 9° F.) for 48 hr., followed by cooling to room temperature in a desiccator. Tests shall be made at room temperature.

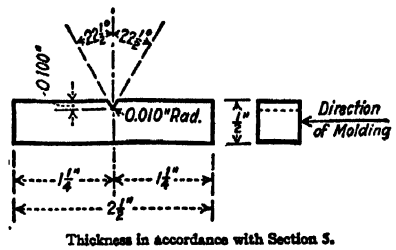
NOTE 1. The type of mold used to produce test specimens has an effect on the results obtained. Cooperating laboratories should, therefore, standardize mold and testing procedure to obtain concordant results.

NOTE 2. In referring to the cutting of the specimens of laminated sheet materials and the application of the load, the following definitions apply:

Flatwise. Load applied to the flat side of the original sheet or plate.

Edgewise. Load applied to the edge of the original sheet or plate.

Izod Test Specimen. For insulating materials other than ceramic materials the specimen shall conform to the dimensions shown in Fig. 297. The notch may be milled, filed or molded provided the bottom of the notch is smooth, straight and free from scratches. For molded material (see Note 1 above) the thickness of the molded specimen, that is, the dimension parallel to the application of the molding pressure and lengthwise of the notch, may be any thickness less than ½ in. agreed upon as representative of the cross-section in which the particular material is to be used. For sheet material the thickness shall be the thickness of the sheet except that it shall not exceed ½ in. Sheet material when thicker than ½ in. may be tested either edgewise or flatwise as specified (see Note 2 above) and when tested flatwise the notch shall be made at the original surface. When the individual specimen is less than ½ in. in thickness, the test piece shall be a composite specimen consisting of a number of individual thin specimens aggregating as nearly as possible ½ in. in thickness. They shall all be accurately aligned with each other and shall be tested edgewise. Specimens of molded insulating material shall be conditioned before testing by drying in an oven at 50° ± 5° C. (122° F. ± 9° F. for 48 hr., followed by cooling to room temperature in a desiccator. Tests shall be made at room temperature.



Courtesy A.S.T.M.

FIG. 297.—Cantilever Beam (Izod) Impact Test Specimen.

Charpy Test Procedure. Five individual determinations of impact strength shall be made. The test specimen shall be supported against the steel blocks so that the blow will be struck at the center of the specimen and on the side opposite the notch for notched specimens. When a composite specimen is used the individual members shall be closely in contact and accurately aligned with each other. It is recommended that a jig be used to insure alignment of notches.

Izod Test Procedure. Five individual determinations of impact strength shall be made. The test specimen shall be rigidly clamped with the center line of the notch on the level of the top of the clamping surface (see Note) and the blow shall be struck on the notched

side. When a composite specimen is used the individual members shall be held closely in contact and accurately aligned with each other when clamped (see Note).

NOTE. It is recommended that a template be used to locate the specimen in the jaws as specified.

Report. The report of the test shall include the following:

(*a*) A statement indicating the type of test, the specimen used and for sheet materials, the direction of testing;

(*b*) For insulating materials other than ceramic materials, the value of energy absorbed in each individual test expressed in foot-pounds per inch of notch, determined by dividing the energy in foot-pounds absorbed in the individual test by the actual dimension in inches along the notch of the specimen broken in each test, whether a single or a composite specimen was used; and

(*c*) The average of the values given in Paragraph (*b*) and the thickness or diameter of the specimen and the number broken in each individual test.

Test 58. Electrical Tests. The A.S.T.M. has similarly standardized a series of tests for these determinations, and for the details the reader is referred to the citations given in the following references:

(*a*) *Dielectric Strength.*
Of sheet, tape and molded insulating materials.[22]
Of solid filling and treating compounds for insulation.[23]
Of electrical insulating oils.[24]

(*b*) *Power Factor and Dielectric Content.*
Of solid insulating materials.[25]

(*c*) *Surface and Volume Restivity.*
Of solid and liquid insulating materials.[26]

(B) SEPARATION OF FINISHED PRODUCT INTO ITS COMPONENT PARTS

SEPARATION OF THE BITUMINOUS MATTER AND DISCRETE AGGREGATE

Bituminized aggregates are separated into their bituminous and discrete components for the combined purposes of ascertaining the percentage and nature of the mineral constituents and for examining the physical and chemical characteristics of the bituminous binder, with the object of its identification or duplication. Two classes of methods are available, depending upon whether the ag-

gregate is associated with an asphaltic or a coal-tar pitch binder.

Various methods have also been described[27] for examining small quantities of such materials, by microanalytical and other procedures. The following, however, constitute the methods generally employed:

Test 59. Methods Suitable for Aggregates Associated with an Asphaltic Binder. Two methods have been devised, one based upon the use of hot extractor, and the other upon the use of a centrifugal extractor.

Test 59a. Hot Extraction Method. This has been standardized as follows:[28]

Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis.

Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enough to be broken up or stirred, so that a representative sample for analysis may be taken. The amount taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a No. 10 sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a No. 10 sieve, 50 g.; where 50 per cent of the aggregate is retained on a No. 10 sieve, 100 g.; and where 75 per cent of the aggregate is retained on a No. 10 sieve, 200 g.

(*a*) *For Analysis of* 10- *to* 30-*g. Samples.* In cases where a 10- to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor illustrated in Fig. 278.

An ordinary fat-free Whatman or S. and S. filter thimble, 60 mm. in length by 26 mm. outside diameter shall be dried for 30 minutes in an oven at 212° F., allowed to cool in a desiccator, and then weighed in a suitable weighing bottle. The weighed sample shall be placed in the thimble and a disc of wool felt or a plug of absorbent cotton shall be placed over the sample in the top of the thimble to distribute the solvent uniformly and prevent splashing. After placing in the extractor, 40 to 50 ml. of carbon disulfide shall

be poured over the sample, whereupon the thimble containing the sample shall be suspended under the condenser by a fine wire bail. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water is circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask.

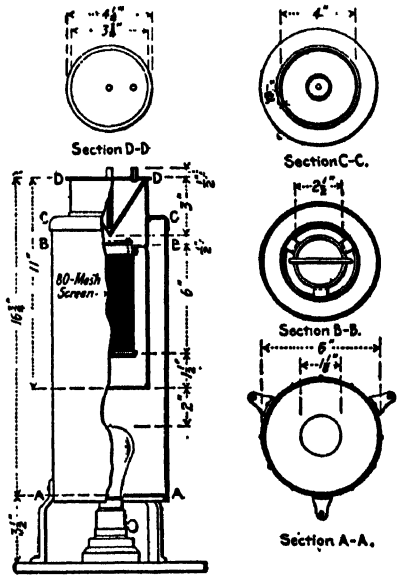
The extraction should be discontinued when the carbon disulfide drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser and other factors. In some cases extraction may be complete in one hour, in others four or five hours may be necessary.

When the solvent comes through clear, the filter shall be removed and washed with a fine jet of carbon disulfide from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed by the carbon disulfide passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to prevent ignition of the absorbed carbon disulfide, and finally to constant weight at 100° C. (212° F.), cooled and weighed.

An aliquot portion of the solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected. The corrected loss in

weight on the original sample represents the percentage of asphalt present.

(*b*) *For Analysis of 50- to 500-g. Samples.* The apparatus for analysis of samples containing coarse aggregate shall be the large extractor shown in Fig. 298, consisting of a large brass cylinder, through the bottom of which projects a 16-candlepower incandescent carbon filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.



Courtesy A.S.T.M.

FIG. 298.—Extraction Apparatus.

A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor by folding once more than in ordinary filtering, or by wrapping it over a form which fits inside the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket.

The basket with contained filter paper shall be dried and weighed. The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another, but to have them mixed together in uniform proportions.

The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 ml. of carbon disulfide poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary

from 3 to 12 or more hours, depending upon the nature of the sample.

To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

The drying and weighing of the basket, burning off of the solution for correction, and calculation of the weight of mineral matter shall be determined as in the foregoing. Note that any water in chemical combination with the mineral constituents is not ascer-

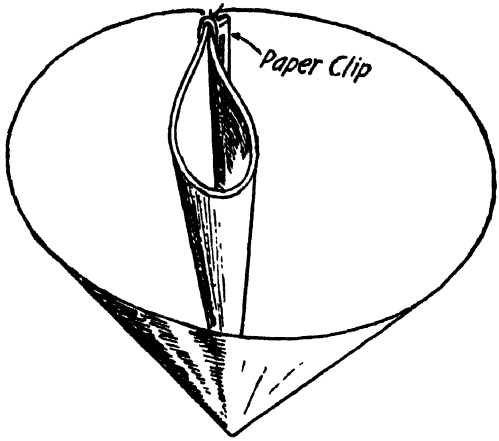


FIG. 299.—Method of Folding Filter-papers for Extraction Tests.

tained by this procedure, but the amount present is usually so small as to be negligible.

Test 59b. Cold Extraction Method. Weigh out 1000 g. of the well-disintegrated sample into an 800 ml. beaker and add 500 ml. carbon disulfide. Stir five or six times during the first 24 hours thoroughly to break up the aggregate, and then let stand quietly for 48 hours, after which the solution shall be carefully decanted and filtered through a series of three 100 ml. funnels, using absorbent cotton as the filtering medium. The aggregate left in the beaker shall be subjected to a second washing, conducted as described. The extracts shall be combined and placed in a quart jar, which shall be tightly covered and allowed to stand quietly for 48 to 72 hours, after which the solution shall be filtered through an asbestos mat in a Gooch crucible to remove the last traces of sediment.

The mineral ingredients shall then be dried and weighed as previously described.

In the case of mastic block and similar bituminized aggregates containing a high percentage of finely-divided mineral matter, there is apt to be a loss of fine material, caused by the carbon disulfide solution creeping behind the folds of the filter paper and over its edge, rather than through the paper. In the case of mastic the following procedure is recommended:[29] Two 32-cm. No. 5 Whatman filter papers, after being dried in an oven and cooled in a desiccator, are counterpoised. These papers are folded together and fastened with a paper-clip in the manner shown in Fig. 299, whereupon they are placed in a glass funnel 7 in. in diameter, the top edge of which is ground flat. The funnel is covered with an 8-in. diameter glass plate in the center of which a 5/8-in. diameter hole has been drilled. To make certain that the plate completely closes the funnel, it is advisable to secure the plate to the funnel with gelatine cement, prepared by dissolving 10 g. of gelatine in 80 ml. water and adding 20 g. glycerin. The mastic block is warmed in an oven and broken into small pieces. Two 75 g. representative samples should be taken and run in duplicate. One sample is introduced into the counterpoised filter and the funnel covered with the glass plate as described. Carbon disulfide is introduced directly into the funnel through a 1500 ml. separatory funnel as shown in Fig. 300. The addition of carbon disulfide may be made automatic, so that when the level of the liquid in the filter falls below a predetermined height,

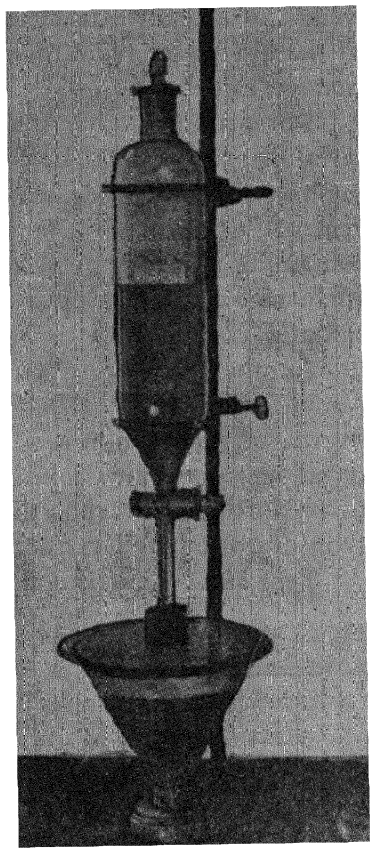


FIG. 300.—Apparatus for Cold Extraction Test.

a fresh supply will be admitted, and this may continue overnight without attention. When the filtrate comes through colorless, the carbon disulfide supply is stopped and the paper allowed to dry, first in air, and finally in an oven at 120° C. for one hour. After cooling in a desiccator, the filter and contents are weighed, the outside paper being placed on the weight-pan as a counterpoise. The weight of asphalt extracted is ascertained by subtracting the weight of the aggregate from the weight of the sample taken, and its percentage calculated.

For products other than mastics, the following weights of material are recommended: surfacings containing aggregate all passing 10-mesh: two 50 g. samples; surfacings containing up to 25 per cent of ¼-in. or ⅜-in. aggregate: two 150 g. samples; surfacings containing up to 25 per cent ¾-in. aggregate: two 300 g. samples; surfacings containing up to 50 per cent ¾-in. aggregate: two 500 g. samples.

Another variation consists [80] in covering the coarsely broken sample with carbon disulfide and allowing same to stand quietly overnight, whereupon it will be found that most of the soluble material has gone into solution. The residue is then washed with 8 to 10 portions of carbon disulfide. Fine mineral matter is removed from the combined extracts by filtering or centrifuging.

Test 59c. Centrifugal Extraction Method. The most efficient apparatus of this type was designed by C. S. Reeve,[81] as illustrated in Figs. 301 and 301*a*. It consists of a ⅕ h.p. vertical motor *a*, capable of making 1100 revolutions per minute at 110 volts, with either direct or alternating current. Its shaft projects into a cylindrical copper vessel *b*, having a concave bottom and draining into the spout *c*. A circular brass plate *d*, 9½ in. in diameter supports an inverted iron bowl *e*, 8½ in. in diameter by 2 5/16 in. high, having a 2-in. circular hole at the top. A brass cup *f* is fastened to the inner side of the bowl, having a circle of ⅛ in. holes for the admission of solvent, and terminating in a hollow axle which fits snugly through a hole in the center of the brass plate *d*. A felt ring *g* ¾ in. wide and about

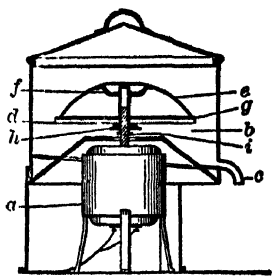
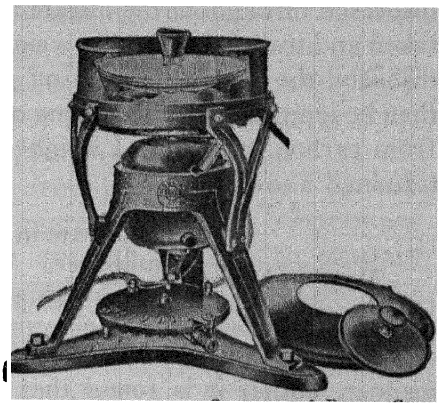


FIG. 301.—Centrifugal Extractor.

0.090 in. thick (cut from No. 80 roofing felt) is firmly pressed against the bowl by the milled nut *h* for which the hollow axle is suitably threaded. The axle in turn fits snugly over the shaft of the motor, to which it is secured by a slot and cross-pin.

Procedure.—The filter ring shall be dried for 30 minutes in an oven at 212° F., allowed to cool in a desiccator and then weighed.

The required quantity (10 or 25 g.) of the disintegrated sample shall be accurately weighed on a tared watch glass or weighing scoop. The sample shall then be placed in the bowl of the rotarex machine, care being taken to evenly distribute the mixture around the periphery of the bowl to prevent vibration during the extraction. The filter ring and bowl plate shall then be placed on the bowl and fastened down fairly tight. The bowl shall then be placed in position in the rotarex machine and from 40 to 50 ml. of carbon disulfide poured into the bowl through the funnel opening. After allowing the material to digest for about 15 minutes, the motor shall be started, slowly at first, and then the speed increased sufficiently to cause the dissolved bitumen to flow from the spout in a thin stream. The dissolved bitumen or filtrate shall be collected in a beaker or other receptacle. When the first charge has drained, the motor shall be stopped and a fresh charge of carbon disulfide added and again allowed to digest for 15 minutes and the machine started as before. This operation shall be repeated from three to five times or until the solvent runs clear from the spout showing all bitumen has been dissolved from the sample, this usually requiring three to four washings.



Courtesy of Braun Corp.

FIG 301A.—Centrifugal Extractor.

When the last addition of solvent has drained off, the bowl shall be removed and placed with the bowl plate and filter ring on a sheet of glazed manila paper and allowed to dry at room temperature. The aggregate shall then be brushed from the bowl onto the paper together with any aggregate adhering to the bowl plate and filter

ring. The aggregate shall then be transferred to a tared watch glass or pan and together with the filter ring dried in an oven at 212° F., cooled in a desiccator and weighed.

In making the correction for the mineral matter suspended in the filtrate, add sufficient carbon disulfide to bring the same to a definite volume and a multiple of 100 ml. The whole filtrate shall then be thoroughly shaken so that the mineral matter is uniformly dispersed throughout the filtrate and an aliquot part of 100 ml. then taken and transferred into a weighed porcelain or silica evaporating dish and the solvent burned off under a hood. The residue shall then be ignited over a gas flame or in a muffle until it is entirely free from carbon, cooled and weighed and the total ash correction determined as follows:

$$\text{Total ash} = \frac{\text{Total filtrate in milliliters} \times \text{Ash in grams recovered in aliquot part}}{\text{Aliquot part in milliliters}}$$

Should there be a considerable amount of ash recovered in this manner, and if it is found that the mineral matter is calcium or other carbonate, it should be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat.

An alternate procedure consists in using a centrifugal tube in the following manner: The apparatus to be used in this test shall be any suitable type of centrifugal machine capable of being rotated rapidly (1800 r.p.m.) surmounted by a head carrying an equal number of metal tube shields, slightly larger than the glass tubes used, and provided with rubber cushions, the whole being encased in a metal shield. In place of glass tubes aluminium or bronze tubes may be used.

Procedure.—The required quantity (10 or 25 g.) of the disintegrated sample selected at random throughout the sample shall be accurately weighed on a tared watch glass or weighing scoop. After weighing, the sample shall be transferred to a numbered and weighed tube. The tube shall be filled with a definite quantity of carbon disulfide approximately three-fourths full and then inserted in the metal tube shield of the centrifuge machine. The tubes shall be so placed in the centrifuge machine as to have equal weights op-

posite each other around the head of the centrifuge. After allowing the material in the tube to digest for about 15 minutes, the centrifuge machine shall be started, slowly at first, gradually bringing it up to full speed and whirling from 5 to 10 minutes. The machine shall then be stopped, the tube removed and the solvent and dissolved bitumen decanted into a numbered flask. The tube shall again be filled with carbon disulfide, mixing the solid matter at the bottom of the tube with the solvent by stirring, the material allowed to digest for 15 minutes, whirled from 5 to 10 minutes, the solvent decanted and the bitumen dissolved as before. This operation shall be repeated from three to five times or until the solvent in the tube, after whirling from 5 to 10 minutes, is clear and colorless.

The tube shall now be removed from the machine and the solvent allowed to evaporate from the aggregate at room temperature and the material then dried in an oven at 212° F. to constant weight, cooled in a desiccator and weighed.

The solvent and dissolved bitumen in the flask shall be rinsed into a weighed porcelain or silica evaporating dish and the solvent burned off under a hood. The residue shall then be ignited over a gas flame or in a muffle until it is entirely free from carbon, allowed to cool in a desiccator and the ash weighed and its weight added to the weight of the mineral matter in the tube. Should there be a considerable amount of ash recovered in this manner, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily the mineral matter in the solution is found to be so small that the difference caused by ignition may be neglected.

Test 60. Method Suitable for Aggregates Associated with Coal-tar Pitch Binder. An apparatus has been devised for this purpose as illustrated in Fig. 302,[82] consisting of a 2-liter copper still fitted to a metal cover of funnel shape, and fastened thereto with six bolts. A flanged glass tube about 1.5 in. in diameter is fastened to the cover by means of a union pipe-joint, as shown. In running the test, 2000 g. of the bituminized aggregate are placed in the still, the cover fastened in place and sufficient sodium carbonate solution (sp. gr. at 60° F. of 1.27 to 1.28) added to bring the surface of the liquid just visible within the glass tube. Gradually apply heat to the

bottom of the still. As the coal-tar pitch liquefies, it rises in globules until it accumulates in a layer in the upper portion of the glass tube. Care should be taken not to heat the liquid higher than necessary to float the pitch. Wash down the reflex condenser with 10 ml. of soda solution to recover adhering oils. Then stopper the upper end of the condenser and remove the clamp from the overflow tube. Heat the bottom of the still rapidly to form a steam pocket below the false bottom, whereupon the liquid pitch will be ejected through the overflow tube and caught in a separate container. Centrifuge the pitch for fifteen minutes to remove all the sodium carbonate solution, and retain it for further examination. If any difficulty is experienced in floating the pitch, as may be the case with old or weathered samples, add 100 g. of solid sodium carbonate at the start of the test. This method will recover all the free carbon with the pitch and separate the mineral aggregate in a clean state.

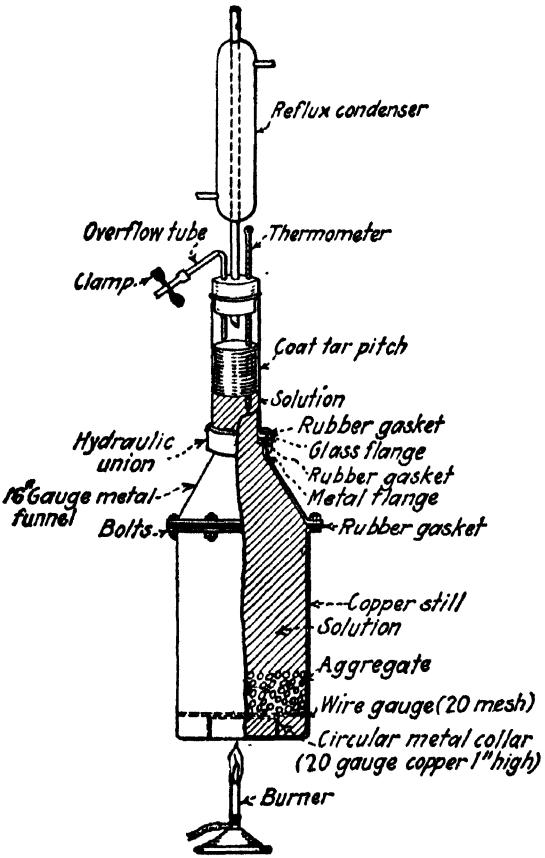


FIG. 302.—Extractor for Coal-tar Pitch Aggregates.

Another method [83] consists in first extracting the bituminized aggregate with carbon disulfide, drying and weighing the residue, which is then boiled for ½ hour with 3 to 5 per cent potassium soap solution, with constant stirring. Decant through a fine mesh screen, wash the residue with water until free from soap, dry the mineral matter and calculate the free carbon by difference.

Another procedure [84] consists in extracting the sample with car-

bon disulfide, drying and weighing the residue. The latter is then tested in two ways, as follows: (1) A portion is heated with 10 per cent HCl, and the CO_2 liberated from any carbonates present in the mineral aggregate is caught and weighed. (2) Another portion is heated with a saturated solution of CrO_3 in concentrated H_2SO_4 and the CO_2 liberated from the carbonates plus that derived from the non-mineral constituents recovered and weighed. The difference between (2) and (1) represents the CO_2 liberated from the non-mineral constituents, which have been found to carry an average of 85 per cent of carbon by weight. The percentage of non-mineral constituents present in the extracted mineral aggregate may be calculated from the following formula:

$$\frac{\text{Per cent (2)} - \text{per cent (1)} \times 0.2727 \times 100}{85}$$

(C) RECOVERY AND EXAMINATION OF EXTRACTED BITUMINOUS MATTER

Test 61. Separation of Bituminous Constituents. The bituminous constituents may be separated from the solvent used for extraction by any of the procedures described in Test 21b. They may thereupon be examined by means of the tests given in Chapter XXXII.

Methods have also been described [35] for examining mixtures of bituminous substances with rubber (e. g. electrical insulating materials, molded articles, rubber substitutes, etc.).

(D) EXAMINATION OF THE SEPARATED AGGREGATE

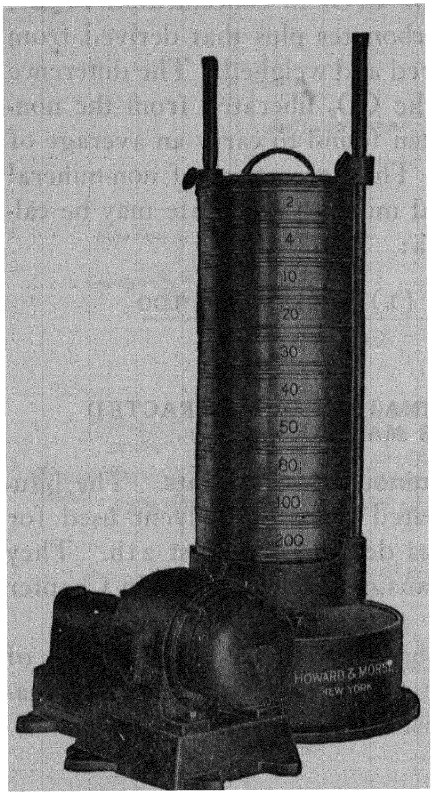
(A) Inorganic Aggregates:

These will include the coarse mineral matter, as well as the fine mineral matter (colloidal) retained on the Gooch filter. After drying, it should be rubbed gently with the fingers to break up lumps and to free any particles of fine dust that might adhere to the larger particles.

NOTE. The presence of any non-mineral matter insoluble in carbon disulfide will be revealed by the discoloration of the mineral particles. In this case, the weight of the latter should be corrected by igniting it until all carbonaceous matter is destroyed, and then reweighing.

The methods which follow have been standardized, and a complete description will be found in the publications cited in the references.

Test 62. Granularmetric Analysis. This is performed by sieving the aggregate through a set of standard sieves of the dimensions given in Table CXII.[36] A convenient mechanical sieving apparatus designed by C. N. Forrest is illustrated in Fig. 303.[37]



Courtesy of Howard & Morse.

FIG. 303.—Mechanical Sieving Apparatus.

A modification consists in washing a suspension of the mineral matter in water through the sieves by decantation.[38]

Various methods have been proposed for the mechanical analysis of sand,[39] mixtures of sand with broken stone or broken slag,[40] also broken stone or broken slag.[41]

(*a*) *Sieving Test.* The following method of test has been proposed [42] for ascertaining the amount of mineral matter or other insoluble matter in asphaltic mixtures that are retained upon a No. 200 (74-micron) sieve: The apparatus shall consist of a 3-in. No. 200 (74-micron) sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes. A 3-in. No. 200 (74-micron) sieve for comparison purposes shall be retained in the laboratory as a standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it, and on the standard sieve, a comparison test, using powdered mineral matter that has a considerable percentage of coarse particles. A reserve stock of such powdered mineral filler should be kept for this purpose.

The sieve shall be dried in an oven at 105° to 110° C., cooled, and then weighed on an analytical balance, the weight being recorded to the nearest 1 mg.

A sample of the asphalt sufficient to yield about 1 g. of mineral matter shall be weighed into a 400-ml. beaker. Then carbon disulfide, carbon tetrachloride or benzol shall be added and the mixture warmed until the asphalt is entirely dissolved. The solution obtained shall be poured through the weighed sieve, and the residue in the beaker shall be washed onto the sieve, using the solvent employed.

TABLE CXII

REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS

Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, Per Cent	Tolerance on Wire Diameter, Per Cent		Tolerance in Maximum Opening, Per Cent
		mm.	in.	mm.	in.		Under	Over	
1	2	3	4	5	6	7	8	9	10
4760 micron(*)	4	4 76	0 187	1 27	0 050	±3	15	30	10
4000 micron . . .	5	4 00	0 157	1 12	0 044	±3	15	30	10
3360 micron	6	3 36	0.132	1 02	0 040	±3	15	30	10
2830 micron.....	7	2 83	0 111	0 92	0 036	±3	15	30	10
2380 micron.....	8	2 38	0.0937	0 84	0 0331	±3	15	30	10
2000 micron.....	10	2 00	0 0787	0 76	0.0299	±3	15	30	10
1680 micron.	12	1 68	0 0661	0 69	0 0272	±3	15	30	10
1410 micron.....	14	1.41	0.0555	0 61	0 0240	±3	15	30	10
1190 micron	16	1.19	0 0469	0 54	0 0213	±3	15	30	10
1000 micron.....	18	1 00	0.0394	0.48	0 0189	±3	15	30	10
840 micron	20	0.84	0.0331	0.42	0 0165	±5	15	30	25
710 micron.....	25	0.71	0 0280	0 37	0 0146	±5	15	30	25
590 micron	30	0.59	0.0232	0 33	0 0130	±5	15	30	25
500 micron.....	35	0.50	0.0197	0 29	0 0114	±5	15	30	25
420 micron.....	40	0 42	0 0165	0 25	0 0098	±5	15	30	25
350 micron.....	45	0.35	0.0138	0.22	0 0087	±5	15	30	25
297 micron.....	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
250 micron .. .	60	0 250	0 0098	0 162	0 0064	±6	15	35	40
210 micron	70	0.210	0 0083	0.140	0.0055	±6	15	35	40
177 micron	80	0 177	0.0070	0 119	0 0047	±6	15	35	40
149 micron.....	100	0.149	0.0059	0.102	0 0040	±6	15	35	40
125 micron.....	120	0.125	0 0049	0 086	0 0034	±6	15	35	40
105 micron....	140	0 105	0 0041	0.074	0 0029	±8	15	35	60
88 micron.....	170	0.088	0 0035	0 063	0 0025	±8	15	35	60
74 micron.....	200	0.074	0.0029	0.053	0.0021	±8	15	35	60
62 micron.....	230	0.062	0.0024	0 046	0 0018	±8	15	35	90
53 micron.....	270	0.053	0 0021	0 041	0 0016	±8	15	35	90
44 micron	325	0.044	0 0017	0.036	0.0014	±8	15	35	90

NOTE. Sieves having larger openings than those included in the table may be specified, but in specifying sieves of larger openings the actual size of the opening (in millimeters or in inches) shall be stated. A tolerance of ±3 per cent on average openings and of 10 per cent in maximum openings shall be permitted.

(*) 1 micron = 0.001 mm.

Sieve Number or Size in inches	Sieve Opening		Wire Diameter		Tolerance, per cent			
	mm.	in.	mm.	in.	Average Opening	Wire Diameter		Maximum Opening
						Under	Over	
No. 100 (149-micron) ..	0.149	0 0059	0 102	0.0040	±6	15	35	40
No. 50 (297-micron)....	0.297	0.0117	0.188	0.0074	±6	15	35	40
No. 30 (590-micron)....	0.59	0 0232	0 33	0.0130	±5	15	30	25
No. 16 (1190-micron)...	1 19	0.0469	0 54	0.0213	±3	15	30	10
No. 8 (2380-micron)...	2.38	0.0937	0 84	0 0331	±3	15	30	10
No. 4 (4760-micron)...	4.76	0 187	1 27	0 050	±3	15	30	10
⅜-in.....	9.5	0.375	2 33	0.092	±3	10	10	10
¾-in.....	19.0	0.75	3 42	0 135	±3	10	10	10
1-in.....	25 4	1 00	4 12	0 162	±3	10	10	10
1½-in.....	38 0	1 50	4 50	0.177	±3	10	10	10
2-in.....	50.8	2.00	4 88	0.192	±3	10	10	10
3-in.....	76.0	3 00	6 3	0.25	±3	10	10	10

The sieve containing the residue shall be washed with the solvent employed, using a camel's hair brush, until free from asphalt and fine mineral matter, then dried and weighed. During the washing, the residue in the sieve shall be covered with the solvent to a depth of approximately ½ in. From the weight of the sample taken, the percentage of ash and the weight of the residue retained on the sieve, the percentage of coarse particles in the mineral matter shall be calculated.

(*b*) *Elutriation Test.* The following methods have been proposed [48] for ascertaining the quantity of clay, loam and silt in gravel, sand and other fine aggregates used in pavements:

The sample as received shall be moistened and thoroughly mixed, then dried to constant weight at a temperature between 100 and 110° C. (212 and 230° F.). The pan or vessel to be used in the determination shall be 12 in. (30.5 cm.) in diameter by not less than 4 in. (10.2 cm.) deep, as nearly as may be obtained.

A representative portion of the dry material weighing not less than 50 times the weight of the largest stone in the sample shall be selected from the sample and placed in the pan which has been dried and accurately weighed. Sufficient water shall be poured into the pan to cover the gravel. The gravel shall be agitated vigorously for 15 seconds with a trowel or stirring rod. After it has settled for 15 seconds the water shall be poured off into a tared evaporating dish, care being taken not to pour off any gravel. This is repeated until the wash water is clear. The washed material shall be dried to constant weight in an oven at a temperature between 100 and

110° C. (212 and 230° F.), weighed, and the net weight of gravel determined.

The percentage of clay and silt shall be calculated from the formula:

$$\text{Percentage of clay and silt} = \frac{\text{Original weight} - \text{weight after washing}}{\text{Original weight}} \times 100$$

For a check on the results, the wash water shall be evaporated to dryness and the residue weighed:

$$\text{Percentage of clay and silt} = \frac{\text{Weight of residue}}{\text{Original weight}} \times 100$$

An alternate procedure [44] consists of the following:*

The pan or vessel to be used in the determination shall be approximately 9 in. (230 mm.) in diameter and not less than 4 in. (102 mm.) in depth.†

The sample must contain sufficient moisture to prevent segregation and shall be thoroughly mixed. A representative portion of the sample sufficient to yield approximately 500 g. of dried material, shall then be dried to a constant weight at a temperature not exceeding 110° C. (230° F.).

The dried material shall be placed in the pan and sufficient water added to cover the sample (about 225 ml.). The contents of the pan shall be agitated vigorously for 15 seconds, and then be allowed to settle for 15 seconds, after which the water shall be poured off, care being taken not to pour off any sand. This operation shall be repeated until the wash water is clear. As a precaution, the wash water shall be poured through a No. 200 sieve and any material retained thereon returned to the washed sample. The washed sand shall be dried to a constant weight at a temperature not exceeding 110° C. (230° F.) and weighed.

The results shall be calculated from the formula:

$$\text{Percentage of silt, loam, clay, etc.} = \frac{\text{Original dry weight} - \text{weight after washing}}{\text{Original dry weight.}} \times 100.$$

* This determination of the percentage of silt, clay, loam, etc., will include all water-soluble material present, the percentage of which may be determined separately if desired.

† A pan satisfying these requirements and which may be readily procured in hardware stores everywhere is known as Nesco Royal Granite Enameled Ware, Step Pan No. 24.

When check determinations are desired, the wash water shall be evaporated to dryness, the residue weighed, and the percentage calculated from the formula:

$$\text{Percentage of silt, loam, clay, etc.} = \frac{\text{Weight of residue}}{\text{Original dry weight}} \times 100.$$

(*c*) *Air-Separation Test.* Various methods have been suggested [45] for separating very fine powders into their component fractions by means of air, including the following:

This method is intended for determining the particle size of particulate substances in absolute units, as far as the particle shape will permit.

The method applies in its entirety to homogeneous materials. In the case of mixtures, the extent of application is limited by the properties of the components of the mixture. The method applies to the range of sizes between the 74-micron (No. 200) sieve and 0.2 micron.

Definitions.

(*a*) *Dispersion.* In statistical work it is measured by the standard deviation. In microscopy it refers to the distribution of the individual particles. Both uses of the word seem too generally accepted to warrant a change in either.

(*b*) *Individual Particle.* Those minute units of matter (of which the material is composed) whose size and shape depend only on the force of cohesion. Such particles cannot be subdivided without separating like molecules that are within the range of the cohesive attraction of one another.[46]

(*c*) *Aggregate.* A group of two or more attached particles.

NOTE. There are three forms of aggregates: namely, ultimate working unit, flocculate, and apparent flocculate.

(*d*) *Ultimate Working Unit.* An individual particle or any group of individual particles that are so firmly held together by forces of adhesion that they remain intact as a group throughout the duration of their application.

(*e*) *Flocculate.* Flocculation occurs only where particles have been incorporated in a liquid or plastic medium. A flocculate is a group of particles held together by a force, apparently arising from interfacial tension. This force of flocculation is relatively weak,

consequently flocculates do not function as large individual particles, and cannot be classified under ultimate working units. Under slight continuously applied force flocculates are constantly breaking down and reforming.

Apparent flocculates as found in gas-floated or air-floated particles show a somewhat similar type of aggregation. Usually the force of attraction between the particles is relatively weak and the particles can readily be dispersed in suitable media. For practical purposes, the more inclusive term, aggregate, is preferable to flocculate for gas-floated or air-floated particles.

(*f*) *Average Diameter.* The diameter of a hypothetical particle which, in some particular way, represents the total mass of particles.

(*g*) *Non-uniformity.* A non-uniform particulate substance is one in which the constituent particles differ from one another in diameters.

Results shall be expressed using one or more of the following average diameters:

Average Diameter	To Be Used:	Formula
d_1	As arithmetical mean	$d_1 = \frac{\Sigma nd}{\Sigma n}$
$\bar{d}_3$	To determine S when ρ is known	$d_3 = \frac{\Sigma nd^3}{\Sigma nd^2}$
$\bar{d}_4$	To determine volume or weight distribution	$d_4 = \frac{\Sigma nd^4}{\Sigma nd^3}$
$\bar{D}$	To determine N when ρ is known	$D = \sqrt[3]{\frac{\Sigma nd^3}{\Sigma n}}$
Δ	To determine S when N is known	$\Delta = \sqrt{\frac{\Sigma nd^2}{\Sigma n}}$
M_g	As geometric mean or median	$\log M_g = \frac{\Sigma n \log d}{\Sigma n}$

Symbols

S_m = specific surface in square meters per gram
S_v = specific surface in square meters per milliliter
N_m = number of particles per gram
N_v = number of particles per milliliter
Σn = number of particles in any given mass
ρ = density of the material
θ = arithmetic standard deviation
θ_g = geometric standard deviation
P.E. = probable error.

Rough Separation into Size Groups. For pigments or other materials which are essentially less than 6 microns proceed directly with the preparation of the mount.

For wide ranges of distribution of particle size in sub-sieve size portions, auxiliary separation into groups of sizes is necessary to facilitate measurement with the microscope. Rough separation is permissible as exact size measurement in microns follows. The following general ranges of particle diameter are proposed for the separation:

A	Down to 24 microns
B	24 to 12 microns
C	12 to 6 microns
D	6 to 0.2 microns

For specific purposes A or D may be further separated.

NOTE. If the sample contains particles larger than the 74-micron (No. 200) sieve, the material shall be separated by sieving wet in a suitable fluid. The portion which passes the sieve may be recovered by evaporation of the fluid. The portion which remains on the sieve may be subjected when dry to a standard sieve test and additional material that passes the 74-micron (No. 200) sieve added to that which is to be tested.

Separation shall be made by air or liquid elutriation or by sedimentation at room temperature. The volume of liquid in the elutriator or sedimentation apparatus should be between 20 and 100 ml. per gram of solid. With finer particles and with material of lesser density the greater volumes should be used. The technique involved in the elutriation or settling is necessarily influenced by the type of material being measured and the dispersing liquids. The size of the vessel and dispersion will be specified in terms of the uniformity obtained. The microscope measurement will be used as a check upon the effectiveness of the separation. For accurate results at least 50 per cent by weight must fall within the boundaries of the suggested ranges set for the elutriation. Of the material falling outside the boundaries not over 5 per cent by weight may fall outside of a range which is one half of the lower limit and twice the upper limit of the range under consideration.

The fluid used in the elutriation or sedimentation shall contain no dissolved solids which would influence the weight determination of a given portion, that is, the fluid shall be 100 per cent volatile. It shall not react chemically with the material being elutriated.

Water, kerosene, alcohols, and the like may be used. It is suggested that the filtering of any elutriator portion should be through alundum to avoid contamination of the sample with paper or pulp fibers. As an alternative, the solid may be recovered from each stage of the elutriator by evaporation of a concentrated slurry, providing the temperatures used for the evaporation do not alter the specific product. The total weight of the portion recovered shall check within 5 per cent the weight of material initially placed in the elutriator.

All limits are specified to insure good microscopic technique. Good elutriation technique for analytical purposes falls well within these limits.

NOTE. Quite obviously, materials containing components of different densities will elutriate or settle in ranges of low uniformity. The present requirements can only be applied when the material meets the following requirements for homogeneity. If the materials can be readily identified under the microscope, they may be measured individually and the results considered as exact, notwithstanding wide differences in density between two materials which may cause lack of uniformity. In other cases, such as cement or coal, where the presence respectively of gypsum and ash constituents may affect the test, these effects may be considered negligible for the normal type of products.

Preparation of the Mount. Any one of a number of methods of preparing the mount may be used, the criterion of suitability being conformity to the following requirements:

The particles shall be essentially in one plane.
The particles shall be free from motion.
The particles shall be dispersed, showing individual grains instead of aggregates and flocculates.
The particles shall not be ground in mounting.
The mount shall be truly representative of the distribution of sizes in the material.
The mounting medium shall be selected to give maximum definition.

(*a*) *Fine Materials, Pigments, etc.* The type of mount that should be used depends on the physical properties of the particles, and for this reason no definite requirements are specified. The general procedure shall be to place the material being tested on a microscope slide, rub it out in a solution of gum or resin in a solvent until the particles are well dispersed. After the solvent has evaporated, the non-volatile constituent of the dispersing agent serves to cement the particles to the slide in the dispersed state.

NOTE. Detailed technique will be found in the work of Dunn[47] and Green.[48] The former uses balsam in xylol, and the latter turpentine and dammar. Other mounting media suggested are 0.5 per cent glycerine in alcohol, rubber cement, styrax in xylol, and the chlorinated naphthalenes and saponin.

(*b*) *Coarse Material, Ground Products, etc.* In the case of coarse materials it is often advantageous to use the so-called dry mount, in which the dispersing agent used is volatile and can be driven off after the material has been dispersed on the slide. The uniformity of the mount when examined microscopically is the best criterion of an acceptable mount.

Direct Observation Method. Direct observations using the Filar micrometer.[49] This type of measurement has been used with fair success and reasonable degree of accuracy on the coarser fraction of particulate substances. The Filar micrometer is an instrument that fits over the ocular of the microscope and permits direct measurement of the particle as viewed on the slide. This method, however, is not recommended for materials in the 0.2 to 6 micron class size.

Projection Method. The projection method[48] which involves throwing the image of the particle on a screen has several distinct advantages. It further magnifies the image, permits focusing through the depth of the mount insuring a proper focus and count of all the particles.

Photomicrographic Method. The photomicrographic method[50] which involves taking a photomicrograph of the particles and subsequently measuring them on a print or by projecting the negative on the screen is also recommended. This method has the very decided advantage of making a permanent record of the sample and permits eye comparisons of two or more samples. In the use of this method it is extremely important that the mount be made according to the method outlined under the method of Green and fulfill the requirements specified there.

Measurement of Diameter. In each of the three methods referred to in the preceding sections, the horizontal diameter shall be measured, unless the particles are very uniform in shape and in that event a diameter which most nearly represents the average shall be selected. For irregularly shaped particles the horizontal diameter splitting the particle in half in one direction consistently maintained has been shown[51] to give fairly consistent and accurate

results. For flat plates it is necessary to consider the third dimension for accurate results. In any case, in reporting results, the diameter measured shall be specified.

Scale Limits for Measurement. If more than 10 per cent by weight is coarser than 6.00 microns, the sample shall be elutriated prior to microscopic measurement. A graded scale for microscopic sizing is given below:

DIAMETER RANGE IN MICRONS

General Case (20 Per Cent or More Under 6 Microns)	Special Case (Less than 20 Per Cent Under 6 Microns)
72 to 60	
60 to 48	
48 to 36	
36 to 24	
24 to 18	
18 to 12	
12 to 9	
9 to 6	
	Same Scale and
6.00 to 5.00	6 to 4.5
5.00 to 4.00	4.5 to 3
4.0 to 3.5	3 to 1.5
3.5 to 3.0	1.5 to 0
3.0 to 2.5	
2.5 to 2.0	
2.0 to 1.8	
1.8 to 1.6	
1.6 to 1.4	
1.4 to 1.2	
1.2 to 1.0	
1.0 to 0.9	
0.9 to 0.8	
0.8 to 0.7	
0.7 to 0.6	
0.6 to 0.5	
0.5 to 0.4	
0.4 to 0.3	
0.3 to 0.2	
0.2	

Two hundred and fifty particles shall be measured in each of three fields, taken at random.

The magnification shall be dependent on the class size being measured, and shall be sufficient to permit the differentiation specified above.

Expression of Results. If 90 per cent by weight fall in any one class size, a frequency curve shall be given.

In the 0 to 6-micron class either a frequency curve or the distribution by weight shall be given.

In elutriated products, weight distribution shall be expressed as follows:

Weight distribution shall be the basis of comparison for all elutriated samples. The weight distribution for each elutriated portion shall be calculated on the basis of spherical particles using the arithmetic mean of the microscopic class interval as the average diameter for all particles in that range. Each of the elutriated fractions shall total in weight distribution to the percentage retained in the elutriator and the composite curve shall be a summation of the distribution values in each range. The degree of overlapping is a measure of the efficiency of the elutriation and it shall not be abnormal in amount.

Test 63. Adsorptive Capacity of Fine Fillers. Most fillers exert more or less of an adsorptive affinity in respect to bituminous substances, whereby a small proportion of the latter remains in combination with the mineral filler upon extraction with solvents. The following test has been proposed as a measure of this "adsorptive capacity": Weigh 5 g. of the filler into a flask and add 30 ml. of a 0.5 per cent solution of a standard asphalt dissolved in c.p. benzol (it is recommended that a 50 to 60 penetration asphalt cement of known origin be used for the purpose). Agitate for ½ hour and filter through paper. Compare the color of the filtrate with a standard series prepared from the same asphalt dissolved in benzol in varying percentages (i.e. 0.1, 0.2, 0.3, 0.4 and 0.5 per cent solutions). The diminution of color is a measure of the adsorptive capacity of the filler.

Test 64. Specific Gravity. Several methods have been standardized [52] for ascertaining the apparent specific gravity of mineral aggregates, fine mineral fillers, pigments, etc. The following procedure [53] is adapted for finding the apparent specific gravity of fine aggregates:

The apparatus shall consist of the following:

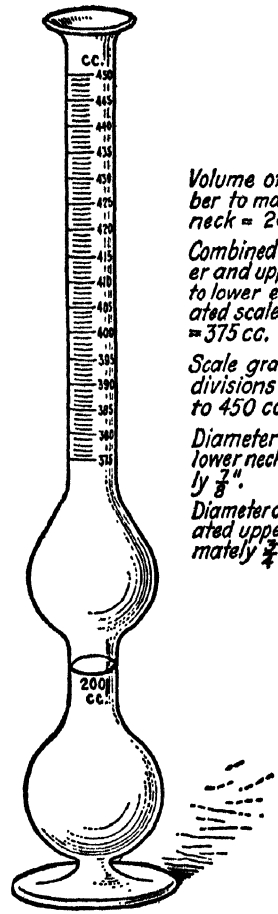
(*a*) *Balance.* A balance having a capacity of 1 kg. or more and sensitive to 0.1 g. or less.

(*b*) *Flask.* A volumetric flask of 500-ml. capacity, calibrated to 0.1 ml. at 20° C. (Fig. 304).

(*c*) *Conical Mold.* A metallic conical mold, 1½ in. in diameter at the top, 3½ in. in diameter at the bottom, and 2⅞ in. in height.

(*d*) *Tamping Rod.* A metal tamping rod, weighing 12 oz., and having a flat circular tamping face 1 in. in diameter.

Approximately 1000 g. of the fine aggregate selected from the sample by the method of quartering shall be placed in a suitable pan or vessel, covered with water, and permitted to stand for 24 hr. The sample shall then be spread on a flat surface, exposed to a gently moving current of warm air, and stirred frequently to secure uniform drying. This operation shall be continued until the sand approaches a free flowing condition. The sand shall then be placed loosely in the conical mold, the surface lightly tamped 25 times with the metal rod, and the mold lifted vertically. If free moisture is present, the cone of sand will retain its shape. Drying with constant stirring shall be continued and tests made at frequent intervals, until the cone of sand slumps upon removal of the mold. This indicates that the sand has reached a surface-dry condition.



Courtesy A.S.T.M.

FIG. 304.—Graduated Flask for Testing Fine Aggregate.

NOTE. The procedure described above is intended to insure that the first trial determination shall be made with some free water in the sample. If the cone of sand slumps on the first trial, the sand has been dried past the saturated and surface-dry condition. In this case a few milliliters of water shall be thoroughly mixed with the sand and the sample permitted to stand in a covered container for 30 min. The process of drying and testing the sand shall then be resumed.

A 500.0-g. sample of the material prepared as described above shall be introduced immediately into the flask and the flask filled almost to the 500-ml. mark with water at a temperature of 20° C.

The flask shall then be rolled on a flat surface to eliminate all air bubbles, after which it shall be placed in a constant temperature bath maintained at 20° C. After approximately 1 hr. it shall be filled with water to the 500-ml. mark and the total weight of water (see Note) introduced into the flask shall be determined to the nearest 0.1 g.

NOTE. If desired, the quantity of water necessary to fill the flask may be determined volumetrically by the use of a burette accurate to 0.1 ml.

The fine aggregate shall be removed from the flask and dried to constant weight at a temperature of 100 to 110° C., cooled to room temperature in a desiccator, and weighed.

The bulk specific gravity shall be calculated from the following formula:

$$\text{Bulk specific gravity} = \frac{A}{V - W}$$

where A = weight in grams of dry sample in air, V = volume in milliliters of flask, and W = weight in grams or volume in milliliters of water added to flask.

The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk specific gravity (saturated surface-dry basis)} = \frac{500}{V - W}$$

The apparent specific gravity shall be calculated from the following formula:

$$\text{Apparent specific gravity} = \frac{A}{(V - W) - (500 - A)}$$

The percentage of absorption shall be calculated from the following formula:

$$\text{Percentage of absorption} = \frac{500 - A}{A} \times 100$$

Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

The following procedure [54] is adapted for finding the apparent specific gravity of coarse aggregates:

The apparatus shall consist of the following:

(*a*) *Balance.* A balance having a capacity of 5 kg. or more and sensitive to 0.5 g. or less.

(*b*) *Wire Basket.* A wire basket of No. 4 mesh wire, approximately 8 in. in diameter and 8 in. in height.

(*c*) *Suitable Container* for immersing wire basket in water and suitable apparatus for suspending wire basket from center of scale pan of balance.

Approximately 5 kg. of the aggregate shall be selected from the sample to be tested by the method of quartering, rejecting all material passing a 3/8-in. sieve. In the case of homogeneous aggregate, all material may be retained on a 1-in. sieve. The sample shall be immersed in water at 15 to 25° C., thoroughly agitated to remove dust or other coatings from the surface of the particles and then allowed to absorb water for 24 hr. It shall then be removed from the water and rolled in a large absorbent cloth until all visible films of water are removed, although the surfaces of the particles still appear to be damp. The larger fragments may be individually wiped. Care should be taken to avoid evaporation during the operation of surface drying. The weight of the sample in the saturated surface-dry condition shall then be obtained. This and all subsequent weights shall be determined to the nearest 0.5 g.

After weighing, the saturated surface-dry sample shall be placed immediately in the wire basket and its weight in water determined. The sample shall then be dried to constant weight at a temperature of 100 to 110° C., cooled to room temperature, and weighed.

The bulk specific gravity shall be calculated from the following formula:

$$\text{Bulk specific gravity} = \frac{A}{B - C}$$

where A = weight in grams of oven-dry sample in air, B = weight in grams of saturated surface-dry sample in air, and C = weight in grams of saturated sample in water.

The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk specific gravity (saturated surface-dry basis)} = \frac{B}{B - C}$$

The apparent specific gravity shall be calculated from the following formula:

$$\text{Apparent specific gravity} = \frac{A}{A - C}$$

The percentage of absorption shall be calculated from the following formula:

$$\text{Percentage of absorption} = \frac{B - A}{A} \times 100$$

Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

The percentage of voids in aggregates (see also Test 7f) may readily be calculated from the specific gravity.[55] The following method has been standardized for determining the percentage of voids in fine aggregate when inundated with water:[56]

The apparatus shall consist of the following:

(*a*) *Balance.* A balance, preferably of the torsion type, having a capacity of 2 kg. or more and sensitive to 0.5 g. or less.

(*b*) *Flask.* A special graduated flask of the type, and conforming to the dimensions, shown in Fig. 304.

A sample weighing about 2 kg. shall be selected which shall be as truly representative of the fine aggregate as possible. It shall be well mixed and spread out on a flat surface and air dried until the surface moisture has evaporated and the sample is free flowing.

About 100 ml. of water shall be placed in the flask. Aggregate from the room-dry sample shall then be introduced slowly into the flask until the flask is filled to the 400-ml. mark on the upper neck. Water shall be added as required to keep the aggregate inundated as the filling proceeds. When the flask is filled to the 400-ml. mark, both water and aggregate shall be at the same level. The aggregate in the flask shall be agitated during the filling operation to insure the elimination of entrained air. The flask and contents shall be weighed to the nearest gram, and the weight of the flask shall be deducted, unless its weight has been counter-balanced on the scale at the time of weighing.

This method requires that the apparent specific gravity of the

fine aggregate be first ascertained in accordance with the preceding method.

The approximate percentage of voids in the inundated fine aggregate shall be calculated from the formula:

$$V = \frac{400 \times \text{sp. gr.} - W}{400\ (\text{sp. gr.} - 1)} \times 100$$

(B) Organic Particles, Fibers, Fillers, etc.:

These constituents may be examined microscopically, or by any of the conventional methods, the scope of which fall outside of the present publication.

CHAPTER XXXIV

EXAMINATION OF BITUMINIZED FABRICS

This caption includes the following groups of products, viz.:

Q—Prepared roofings.
R—Composition shingles.
S—Deck and porch coverings.
T—Bituminized fabrics for constructing built-up roofs.
U—Bituminized fabrics for constructing waterproofing membranes.
V—Electrical insulating tape.
W—Waterproof papers for wrapping and packing.
X—Waterproof papers for insulating against heat or cold.
Y—Felt-base floor coverings (surfaced with linseed oil and pigment composition).
Z—Expansion joints for pavements.

These are constructed as shown in Table CXIII, where the index *a* indicates that asphaltic compositions have been used, and *t* signifies that coal tar (pitch) *et al.*, have been used.

The finished products falling in this class include sheet roofings, floor coverings, waterproof membranes, sheathing and insulating papers, expansion joints involving the use of woven or felted fabrics, electrical insulating tape, and certain types of wall board. As these are constructed in many different ways, it will obviously be impracticable to describe in detail the analytical methods applicable to each. The ones which follow have been devised specifically for examining prepared roofings,[1] but with these as a starting-point, others may readily be evolved for testing floor coverings, waterproof membranes, sheathing and insulating papers, etc.

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT

Test 65. Weight per Unit Area. The following methods have been standardized:

(*a*) *For Saturated Felted and Woven Fabrics.*[2]

The width of each roll taken shall be measured to the nearest

TABLE CXIII

	Paper		Burlap		Duck		Light Cotton Fabric		Rag-Felt		Asbestos Felt		Burlap and Rag or Asbestos Felt		Paper and Light Cotton Fabric	
	a	*t*	*a*	*t*	*a*	*t*	*a*	*t*	*a*	*t*	*a*	*t*	*a*	*t*	*a*	*t*
Single Layered:																
Saturated only	WX	WX	U	U	...	...	...	...	TUY	TU	T	...	...	...	...	...
Coated only (one or two sides)	W	....	U	U	S	...	...	...	.....	.....	...	...	...	...	...	...
Saturated and coated	X	....	U	U	QS	...	V	...	QR	....	Q	...	...	...	...	...
Laminated (Bituminated):																
Layers saturated only	....	....	Z	...	...	...	...	...	Z	Q	QT	...	UZ	U	...	...
Layers saturated and coated	....	....	...	...	...	...	...	...	Q	....	Q	...	Q	...	...	...
Layers unsaturated	W	W	...	...	...	...	...	...	.....	.....	...	...	...	...	W	W
One layer unsaturated and others saturated	W	W	...	...	...	...	...	...	.....	.....	...	...	...	...	W	W

NOTE. Letters in heavy type indicate the more important groups of products.

1/32 in. The minimum and maximum width so obtained shall be reported.

Each roll taken shall be weighed intact to the nearest 1/4 lb. and the minimum and maximum gross weight shall be reported.

Each roll sampled shall be stripped. All the wrappers and packing material shall be weighed together to the nearest 1/4 lb. and the average weight thereof per roll shall be reported. Each roll shall then be unwound, observing the workmanship and finish while so doing. The length and width of each roll shall thereupon be measured to the nearest 1/4 in. and the square feet of material contained in each roll calculated.

The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest 1/4 lb. In the case of felted fabrics, calculate the weight of each roll in pounds per 100 sq. ft. and report the maximum and minimum, likewise recording the average for the rolls sampled, which shall be regarded as the

corresponding weights of the entire shipment. In the case of woven fabrics, calculate the weight of each roll in ounces per square yard, and report the minimum, likewise recording the average for the rolls sampled, which shall be regarded as the corresponding weights of the entire shipment.

From the rolls examined, select the one whose weight per unit area is nearest the average weight per unit area of the lot. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A section measuring 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material, any detached particles belonging to each 30-in. section shall be retained. The width of each section shall be measured to the nearest $\frac{1}{32}$ in. Each section, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per sq. yd. shall be calculated from the formulas:

$$\text{Pounds per 100 sq. ft.} = 1.0582 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

$$\text{Ounces per sq. yd.} = 1.5238 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

This must check within 1 per cent of the average weight per unit area of shipment. If the sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE. As a referee method or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled as above.

If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. sections taken. All the material thus removed shall be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of detached comminuted surfacing shall be calculated in pounds per 100 sq. ft.

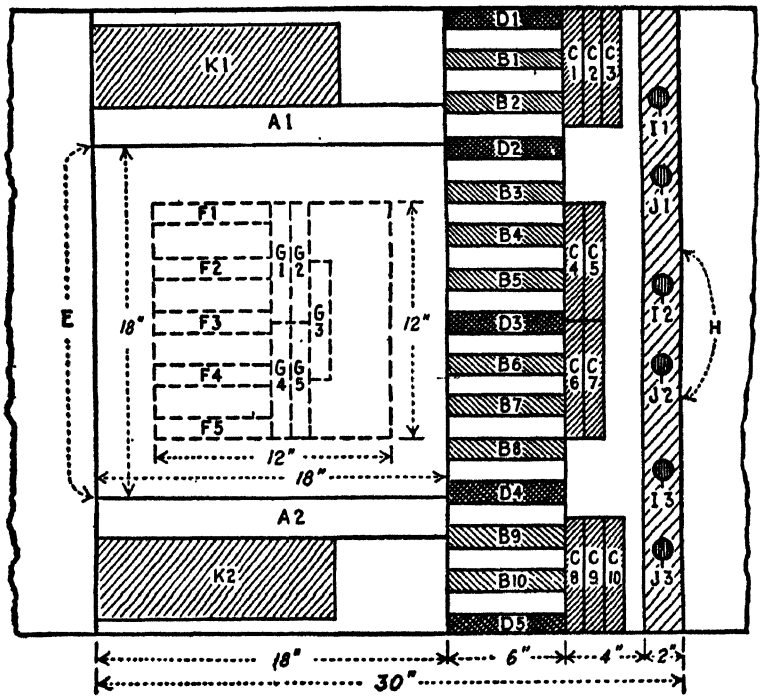
The 30-in. section selected for examination shall be cut into sections for further tests in the manner shown in Fig. 305.

(b) For Smooth-roll and Mineral-Surfaced Roll-Roofing[3]

Each roll selected shall be weighed to the nearest ¼ lb. The wrappers, packing material, nails, and cement from each roll shall be weighed together to the nearest ¼ lb. and the average weight thereof per "square" shall be recorded. The quantity and character of nails and cement shall be recorded.

Each roll shall be unwound and the workmanship and finish of the roofing observed. Record the weight of any loose surfacing that may fall off in so doing.

The length and width of each roll shall be measured to the



Courtesy A.S.T.M.

FIG. 305.—Sampling Bituminized Fabrics.

nearest ¼-in., and the square feet of material contained in each roll shall be calculated. From these values, the average area of the rolls examined shall be calculated.

From the above, the net weight, exclusive of loose surfacing, of roofing per 108 sq. ft. contained in each roll shall be calculated and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

The average weight per 108 sq. ft. for the rolls examined shall

be calculated. This shall be regarded as the average weight per 108 sq. ft. of the lot..... (1*a*)

From the rolls examined, the one whose weight per 108 sq. ft. is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled, and with a knife and straight edge, the sheet shall be cut across at right angles to the edges. A section measuring exactly 30 in. in the direction of the roll's length shall then be removed. The width of this section shall be measured to the nearest 1/32 in. and weighed, neglecting any loose surfacing. The weight in pounds per 108 sq. ft. shall then be calculated from the following formula:

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of 30-in. section in ounces}}{\text{Width of 30-in section in inches}} \times 32.4 \quad (1b)$$

The weight so determined shall check within 1.5 per cent of the average weight per 108 sq. ft. of the lot (1*a*). If this sample fails to check, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be kept for further examination.

NOTE. As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled.

(*c*) *For Mineral-surfaced Shingles*:[8]

Each bundle selected shall be weighed to the nearest 1/4 lb. The packing material shall be weighed to the nearest 1/4 lb. and the weight recorded separately for each bundle. The average weight of packing material per "square" shall be recorded.

The shingles in each bundle, "*a*," shall be counted and the workmanship and finish of the shingles shall be observed. Record the weight of any loose surfacing that may fall off in so doing.

From the above the net weight of each bundle, exclusive of loose surfacing, shall be calculated, "*b*." The dimensions of two representative shingles shall be accurately measured and the net area of material in each shingle calculated in square feet, "*c*."

The area in square feet of material contained in each bundle ($a \times c$) shall be calculated. The weight of roofing material per 108 sq. ft. contained in each bundle $\left(\frac{b}{a \times c} \times 108\right)$ shall be calculated, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

The average weight (d) per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment..... ($1a$)

The average weight per shingle $\left(\frac{c \times d}{108}\right)$, shall be calculated ($1b$)

From the bundles selected, a definite number of representative shingles, whose weight shall fall within 1.5 per cent of the average weight ascertained in ($1a$) shall be taken for further examination. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE. As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, then a similar sample shall be taken and examined separately from each bundle of shingles sampled.

Test 66. Thickness. The thickness of each specimen shall be measured at 10 equally spaced points, 1 in. from a cut edge, with a micrometer gage having flat bearing surfaces at both contact points of not less than ½ in. in diameter and preferably of the spring-and-dial type. The readings for each roll sampled shall be averaged and the minimum, maximum and average thickness calculated to the nearest 0.001 in.

Test 67a. Tensile Strength.

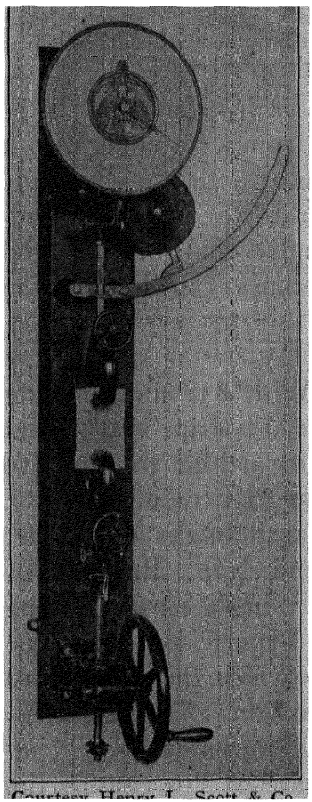
(*a*) *For Felted Fabrics and Papers*: With a trimmer having a stop attached exactly 1-in. behind the blade, 10 strips shall be cut with the fiber grain, as shown at B-1 to B-10 in Fig. 305, and 10 strips across the fiber grain, as shown at C-1 to C-10 in Fig. 305. Each strip should measure 6 in. and have parallel sides 1 in. apart (within 1/32 in.) with the edges cut straight and clean. Both sets of strips shall be tested at 70° F. (21.1° C.), using a tension testing machine such as the Scott Strength Tester (Type F, 100-lb. capacity), the Perkins Strip Tester, or equal, in which the clamps are attached to swivels free to move in any direction. The test strips shall be gripped 1½ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased by causing the lower clamp of the machine to travel at a uniform speed of 12 in. per minute. If any strip breaks nearer than ½ in. to either clamp, the reading shall be disregarded, and an additional strip shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these

results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated.

An instrument for ascertaining the tensile strength is illustrated in Fig. 306, consisting of a pendulum type of machine. The pointer remains in position when the specimen breaks, and is reset by means of the handle suspended from the curved quadrant.

(*b*) *For Woven Fabrics*: This test shall be conducted as described in Test 79c applicable to the desaturated fabric.

Test 67b. Bursting Strength. The Mullen Tester is used for this purpose as illustrated in Fig. 307. This instrument has a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber is filled with glycerin and the test specimen is held in position over the diaphragm in a clamp having a circular hole 1 sq. in. in area, so that the diaphragm will force the material into the hole when pressure is applied under the diaphragm. The pressure is indicated on a dial graduated in 0.5 lb. divisions and during the test is increased at the uniform speed of 2 lb. per second at a uniform temperature of 77° F. Ten tests shall be averaged.

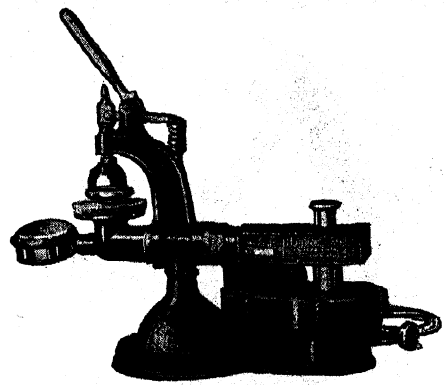


Courtesy Henry L. Scott & Co.

FIG. 306.—Tensile Strength Tester.

Test 67c. Tearing Strength. This is determined by means of the Elmendorf Tester illustrated in Fig. 308,[4] consisting of a heavy sector *A* carrying a dial graduated from 0 to 100, mounted on ball-bearings on the support *B*. The instrument is leveled by the screw *C* in the base *D*. When the instrument is level, a white mark *E* on the sector will be opposite the end of the spring-stop *H* when the sector is free to swing. To test for zero, push the sector *A* to the left until the spring-stop *H* engages in the right-hand edge of the sector. Set the pointer *K* against the pointer stop *L*, which is fastened to the base by the

screw *M*. Release the sector by depressing *H* as far as it will go and hold it down until the sector has swung once in either direction, and then release the spring-stop. Note the reading of the pointer *K*, and if not at zero, adjust the position of the pointer stop *L* by means of the screw *M*, until the pointer on further trial comes to zero. Insert a sample *P*, 6.3 cm. (2.5 in.) in width and at least 6.3 cm. (2.5 in) in length, cut with straight edges, in the clamps *N*, and set sector *A* and pointer *K* as in the zero test, then depress the cutter lever *O* to low position and release, thereby cutting the strip a distance of 1.7 in. across the 2.5 in. width. The specimen should be tested at a temperature of 75–79° F. Depress the spring-stop as far as it will go and hold it down until the sector has swung once in each direction. The distance swung by the sector is indicated by the position of the pointer, which is recorded as a measure of the tearing strength. The material is tested separately with and against the fiber grain. The author has found that in the case of composition roofings, the tearing strength against the fiber grain remains substantially unaffected as the specimen ages, whereas the tearing strength with the fiber grain gradually increases with age, until it eventually exceeds the former.



Courtesy B. F. Perkins & Sons, Inc.

FIG. 307.—Bursting Strength Tester.

This test has been standardized as follows:

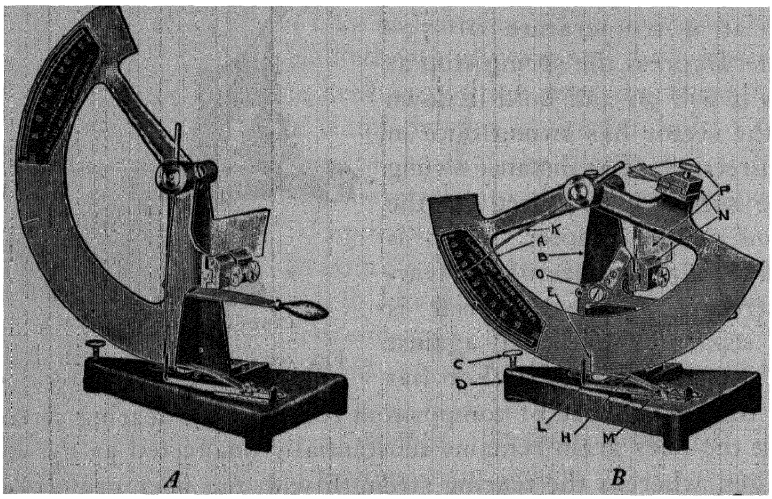
The testing machine shall be of the pendulum impulse type so designed as to produce a tear approximately 4.2 cm. (1.66 in.) long. The knife mounted on the machine to cut the slit for the tear shall be maintained sharp.

The specimens shall be taken from the original samples and shall be cut 6.3 cm. (2.5 in.) in width and at least 6.3 cm (2.5 in) in length. Enough specimens shall be cut so that at least five readings in the machine direction and five readings in the cross-direction, if possible, can be obtained for each original sample.

Enough specimens shall be torn so that the readings on the machine shall be not less than 10 nor more than 20 g., unless one

paper alone yields a reading greater than 20 g., in which case only one paper shall be torn at one time. The specimens shall be so arranged that an equal number of tears originating from opposite edges of the specimens will be produced. Readings obtained when a tear deviates more than 6.3 mm. (0.25 in.) from a straight line, shall be rejected. The readings obtained shall be multiplied by the intrument constant corresponding to the number of sheets torn.

The tearing strengths obtained on the machine-direction specimens and on the cross-direction specimens shall be reported separately in grams. The average, the maximum, and the minimum



Courtesy Thwing Instrument Co.

FIG. 308.—(*A* and *B*).—Tearing Strength Tester.

tearing strengths shall be reported for the machine-direction specimens. If possible, similar results shall be reported for the cross-direction specimens.

Test 68. Pliability. Two methods have been proposed for this determination, as follows:

(*a*) *Mandrel Test*: This has been standardized as follows:[5]

With the trimmer, five 6-in. strips shall be cut with the fiber grain, as shown at D-1 to D-5 in Fig. 305, each 1-in. in width, and immersed in water at 77° F. (25° C.) for from 10 to 15 minutes. These strips shall be bent through 180 deg. at a uniform speed, in exactly two seconds, around a mandrel the diameter of which shall

be as follows: The first, around a mandrel 25 mm. in diameter; the second, around a mandrel 20 mm. in diameter; the third, around a mandrel 15 mm. in diameter; the fourth, around a mandrel 10 mm. in diameter, and the fifth, around a mandrel 5 mm. in diameter. The test may also be made at 32° F. (0° C.). The pliability shall be expressed numerically from 1 to 10 as follows:

(1) Cracks entirely through the sheet on the 25-mm. mandrel.
(2) Cracks part way through the sheet on the 25-mm. mandrel.
(3) Cracks on the 25-mm. mandrel.
(4) Cracks on the 20-mm. mandrel.
(5) Cracks on the 15-mm. mandrel.
(6) Cracks on the 10-mm. mandrel.
(7) Cracks on the 5-mm. mandrel.
(8) Cracks when bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel.
(9) May be bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel in one direction without cracking, but will crack when bent through 360 deg. in the opposite direction.
(10) May be bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel in one direction and then through 360 deg. over a 1/16-in. (1.588-mm.) mandrel in the opposite direction without cracking.

The readings for each roll shall be averaged separately, and from these results the minimum, maximum and average pliability of the felt shall be calculated.

A convenient instrument for this purpose is illustrated in Fig. 309.

Saturated-felts are usually bent around a 1/16-in. mandrel.

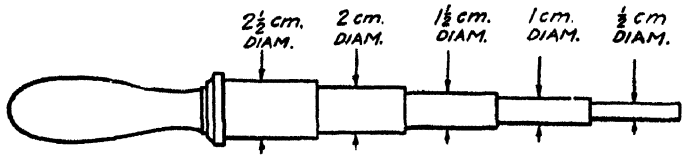


FIG. 309.—Mandrels for Testing Pliability.

Smooth-roll roofings and mineral-surfaced roll-roofings are tested as follows:

With a photographic trimmer, ten strips shall be cut from a sample of roofing, each 1 in. in width and 8 in. in length, five strips in the direction of the fiber grain, and five across the grain. These strips shall be immersed in water at 77° F. (25° C.) for 10 to 15

minutes, after which they shall be removed from the water and immediately bent through an angle of 90 deg. over a rounded corner of ½ in. radius in exactly two seconds without exerting any strain upon the test strip other than that required to keep it in contact with the rounded corner and to avoid kinking.

A block of wood 3 in. square by 2 in. thick with rounded corners of the dimension specified shall be used in making this test.

The strips of roofing, exposure side up, shall be held by hand tight against the upper 2-in. face of the block. The projecting end of the strip shall then be bent over the rounded corner of specified radius with the other hand.

Surface ruptures aggregating more than ⅛ in. in length in any direction shall be considered a crack.

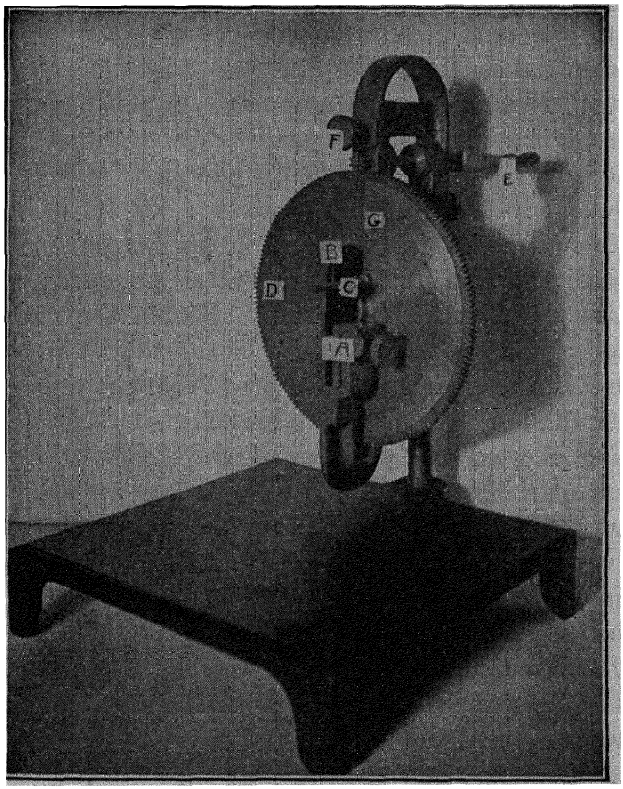
(*b*) *Reeve and Yeager Tester*: This is a more accurate tester, illustrated in Fig. 310.[6]

The essential parts of the apparatus are : the clamp, *A*, holding the strip of roofing under test, and the large wheel, *D*, carrying the pin, *C*, 1 inch distant from its center and projecting perpendicularly from its surface. The clamp, *A*, is designed to hold the lower 2 in. of a roofing specimen 4 in. long and 1 in. wide. This clamp is located directly in front of the large wheel so that the back of the clamp, the top of which is rounded to a 3/16-in. radius, serves as a mandrel located on the central line of the bearing on which the large wheel revolves. Thus placed, the pin, *C*, projecting perpendicularly from the face of the large wheel, and the rounded back of the clamp, are parallel.

When a strip of roofing, *B*, is held in the clamp, as shown, rotation of the large wheel brings the pin into contact with that part of the strip projecting above the clamp, and by continuing the rotation, the pin, bearing on the strip, causes it to bend about the 3/16-in. rounded back of the clamp. The limit of the rotation is reached when the strip has been bent double over the back of the clamp, practically equivalent to 180 deg. of bend.

The rotation of the large wheel and the consequent bending is readily accomplished and controlled by turning the handle, *E*, operating the small pinion gear in mesh with the large wheel. The rate of turning is controlled by a metronome to a speed equivalent to bending the specimen through 180 deg. in thirty seconds.

For convenience in measuring the angle through which the strip is bent, the protractor, *G*, graduated in degrees, is fixed to the wheel, *D*, in a suitable position. In starting a test, the pin is brought in contact with the projecting strip of roofing when it is in a vertical position and the pointer shown just below the set screw, *F*, is adjusted to indicate zero. This feature provides for roofings of dif-



Courtesy A.S.T.M.

FIG. 310.—Apparatus for Measuring the Pliability of Roofing.

ferent thicknesses requiring slightly different initial positions of the large wheel.

As will be seen from Fig. 310, the upper surface of the strip at the point where the bending occurs is at all times clearly visible, so that the development of a break in the coating is readily discerned. When this occurs, the motion of the machine is stopped and the

angle of bending read by the indicator and protractor. This value is recorded as a measure of the pliability of the roofing.

To secure reproducible test conditions, the necessary temperature control is obtained by means of a water bath. The large wheel and clamp are carried on a vertical support, so designed that they can be lowered into the water bath, which is placed on the broad base of the machine and which is made high enough to provide a water level just readily inserted and removed without raising apparatus from the control bath.

Test 69. Resistance to Dampness. Accurately cut a strip of bituminized fabric 18 in.×18 in., and weigh. Remove the detached mineral particles from both sides of the sheet with a moderately stiff brush, and reweigh (area equals 2¼ sq. ft.). Suspend in a tight box containing sufficient water at the bottom to saturate the air with moisture. Cover tightly and allow the specimen to remain in the moist air for 100 hours at 77° F. As the moisture enters more readily through the cut edges of the sheet than through the surface itself, 6 in. should be trimmed from the edges at the termination of the test, leaving a strip measuring exactly 12 in.×12 in., representing the central portion of the original specimen, and weighing ⅘ of the latter. Ascertain the weight, thickness and tensile strength of the 12×12 portion at the end of the test, and calculate any variation in percentage from the original figures. The increase in weight should be figured on the basis of the original material *including* the detached mineral matter.

Test 70. Water Absorption. A section 18-in. square, as shown at *E* in Fig. 305 shall be cut from each specimen, weighed, and completely immersed in distilled water at 77° F. (25° C.) for twenty-four hours. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen, by multiplying by 2¼. The percentage increase in weight represents the water absorption. The minimum, maximum and average for the shipment shall be calculated.

Five strips shall be cut with the fiber grain as shown at F-1 to F-5 in Fig. 305; five other strips shall be cut across the fiber grain as shown at G-1 to G-5 in Fig. 305 and the strength shall be redetermined. The decrease in strength in percentage shall be calculated after the specimen has been subjected to water.

Test 71. Resistance to Heat. The following methods have been proposed:

(*a*) *For Asphalt-saturated Fabrics*: From each specimen, 12 by 6-in. strips shall be cut at K-1 and K-2 in Fig. 305, care being taken not to disturb any of the detached surfacing. Each strip shall be weighed and suspended in the center of an air-oven maintained at 221° F. (105° C.) ±5° F. (3° C.) with a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be kept in the oven for exactly five hours, then cooled and removed carefully, and each specimen weighed. The average loss shall be calculated as a percentage and the average percentage of moisture deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

(*b*) *For Coal-tar Saturated Felt Only:* From the rolls making up the sample, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. ±5.0 g. These strips then shall be rolled up and placed in the wire basket of the extraction apparatus described in Test 59. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent.

The carbon disulfide extract containing the bitumen shall be transferred to a 500 ml., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water-cooled condenser. A —2 to +80° C. thermometer shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulature. Distillation for the removal of carbon disulfide shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the

thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame.

When the contents of the flask are sufficiently cool to be poured without appreciable vaporizing, 100 g. ±0.1 g. shall be distilled in accordance with Test 16b(*c*).

Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation.

(*c*) *For Asphalt Roll-roofings and Shingles*: Two strips shall be cut from the sample of roofing or shingles, each approximately 8 by 8 in. They shall be weighed and suspended vertically in the same direction as the material would be applied to the roof, in the center of an air-oven maintained at 176° F. (80° C.) ±5° F. (3° C.) with a thin wire fastened through holes punctured near one edge. The internal dimensions of the oven shall not be less than 12 by 12 by 12 in. An electrically heated oven is recommended. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The strips shall be maintained at the prescribed temperature for exactly two hours, then cooled in a desiccator and each specimen weighed. The average loss shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering or absorption of the coatings; also sliding of granular surfacing.

Test 72. Electrical Tests. The following tests have been standardized, a full description of which will be found upon referring to the original sources:

Dielectric Strength.
Of sheet insulating materials.[7]
Of laminated sheet insulating materials.[8]
Of adhesive insulating tape.[9]
Of varnished cloths and varnished cloth tapes.[10]

Test 73. Special Tests Applicable to Insulating Tape. The following tests have been standardized for adhesive insulating tape:[9]

(*a*) *Tensile Strength.* This is measured at 77° F. by sepa-

rating a specimen at the rate of either 12 or 20 in. per minute, the initial distance between the jaws of the testing machine being 12 in.

(*b*) *Adhesion.* The adhesion between adjacent layers of the tape shall be determined as follows: A specimen 23 in. in length shall be removed from the sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One end of the specimen shall be inserted in the slot of the mandrel described below and 2 in. of the tape shall be wound on the mandrel. A weight of 10 lb. per inch of width shall then be attached to the end of the specimen and 19 in. of the tape shall be wound on the mandrel at a uniform rate of 12 in. per minute. The tape shall be allowed to remain 3 min. with the weight attached, after which a weight of 4 lb. per inch of width of tape shall be substituted for the weight of 10 lb. per inch of width, and the tape allowed to unwind. After 2 in. have unwound, the rate of unwinding shall be not greater than 15 in. in 1 min. The temperature of the room and the temperature of the tape shall be not less than 20° C. (68° F.) nor more than 22.2° C. (72° F.), except that tape meeting the adhesion test when tested at a higher room temperature shall not be rejected. The relative humidity at the temperature tested shall not exceed 80 per cent.

The mandrel used shall be ¼ in. in diameter with a slot approximately 1/16 in. in width and long enough to accommodate the full width of tape, and shall be mounted in a level position in ball bearings of the Fafnir Bearing Co., Catalog No. 36, Serial No. 30, extra small, or equivalent ball bearings. The mandrel shall turn freely under a weight of ¼ oz., suspended from a cotton thread wound in a single layer on the center of the mandrel.

(*c*) *Oven Test.* After a specimen of tape has been exposed to dry air at a temperature of not less than 99° C. (210.2° F.) nor more than 101° C. (213.8° F.) for 16 hr., and after a rest period at room temperature of not less than 1 nor more than 12 hr., the test specimen shall withstand the test prescribed in (*a*), except that the weight applied to unwind the tape shall be 3 lb. per inch of width in place of 4 lb. per inch of width.

(*d*) *Tackiness.* The tape shall show tackiness, that is, ability to stick to itself after light contact has been made, in the following combinations: (1) front to front, (2) back to back, and (3) back

to front. Two 8-in. specimens shall be cut from each sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One specimen shall be placed lightly on a clean horizontal surface, then the second placed evenly upon the first. No weight or pressure other than the weight of the specimen shall be applied to the tapes. The two specimens shall be picked up from one end and then stripped slowly from each other starting at the same end. Tackiness in at least two of the combinations (1), (2) and (3) specified shall be such that the point of separation remains approximately in the same horizontal plane as the hands that pull the tape apart. In the back to back combination the specimens shall show some indication of tackiness by the point of separation traveling along the tapes as the hands pull the tape apart.

(B) SEPARATION OF FINISHED PRODUCT INTO ITS COMPONENT PARTS

Separation of Bituminous Matter, Mineral Matter and Fibrous Constituents

Test 74. Moisture. From each specimen, 2-in. strips shall be cut, as shown at A-1 and A-2 in Fig. 305. The 2-in. strips shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and distilled with 100 ml. of a coal-tar distillate of which 5 to 10 per cent boils below 100° C. (212° F.) and of which at least 90 per cent distills up to 180° C. (356° F.), in an apparatus of the form and under the conditions prescribed in Test 25b. The flask containing the solvent and sample shall be heated in a paraffin bath at a temperature of 302 to 338° F. (150 to 170° C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the average percentage of moisture in the fabric, as received, shall be calculated.

Test 75. Analysis of Saturated Fabrics (Single-layered). The following method has been proposed:[11]

From each roll sampled, a 2-in strip (within 1/32 in.) shall be cut across the specimen as shown at H in Fig. 305.

Each strip shall be extracted with carbon disulfide in an extrac-

tor shown in Fig. 298, the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225° F. (107.1° C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant, as determined by two consecutive weighings taken not less than ten minutes apart, which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in the following paragraph.

The following colorimetric method shall be used to ascertain the carbonaceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(*a*) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the fibers shall be accurately weighed into a flask and diluted to exactly 100 ml. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(*b*) A distilled coal tar, having approximately 10 to 25 per cent of insoluble carbonaceous matter, shall be procured and the carbonaceous matter extracted with benzol until it is free from soluble matter; then dried at a temperature of 225° F. (107.1° C.) to constant weight. One gram of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 ml. at room temperature with a starch solution of a consistency sufficient to carry the carbonaceous matter into temporary suspension. (A 12.5 per cent solution is recommended.)

(*c*) The liquid carrying the fibers, obtained as described in Paragraph (*a*), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (*b*), and from time to time a field prepared from a drop of the well agitated mixture shall be examined under a microscope at 100 diameters magni-

fication until the color exactly matches a field prepared from the desaturated fabric under examination, when both are reviewed side by side under parallel conditions. The end-point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

(*d*) The weight of moisture-free fabric shall be corrected by deducting the weight of adhering carbonaceous matter.

The total comminuted surfacing plus any filler present in the bituminous saturant represents the sum of the detached comminuted surfacing, the amount brushed off from the surface of the desaturated fabric, plus the amount recovered upon evaporating and igniting the bituminous extract obtained as above.

The weight of bituminous saturant represents the difference between the weight of the original fabric, and the sum of the weights of the moisture-free desaturated fabric, plus moisture, plus the total comminuted surfacing and filler present.

Ash. A representative sample shall be secured by cutting from each strip of desaturated felt a piece about ½ in. in diameter as shown at I-1, I-2, and I-3 (Fig. 305'). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

NOTE. As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum and average of ash present shall be calculated on the basis of the moisture-free felt.

Test 76. Analysis of Saturated and Coated Fabrics (Single-layered). Since the fabrics and bituminous matter may be assembled in many different ways, the resultant products are too numerous to itemize. It is impractical, therefore, to give analytical methods applicable to each. The ones which follow have been devised specifically for examining prepared roofings and composition shingles composed of roofing-felt, saturated and coated on both sides with asphalt, and surfaced on the top with either powdered or granular mineral matter, and on the under side with powdered min-

eral matter. These methods are typical ones, and with slight modifications and a little ingenuity may be adapted to other forms of bituminized fabrics.[12]

Asphalt roll-roofings and shingles may be divided into two general types as shown in Fig. 311.

Type X represents a layer of felt, saturated and coated with bituminous matter, having the surface coatings dusted with fine mineral matter (e. g., talc). This type embraces what is commercially known as "smooth-surfaced roll-roofing."

Type Y is similar to Type X, but with a top surfacing of either: (1) coarse mineral matter (e. g., granular slate) embracing what are commercially known as "mineral-surfaced roll-roofing" and "mineral-surfaced shingles"; or (2) moderately coarse mineral matter (e. g., granular talc) embracing what is commercially known as "roll-roofing surfaced with granular talc."

The mineral matter, bituminous matter and fibrous matter may be distributed in the following manner:

A—Mineral Matter

1. Detached	*Fine Mineral Matter* (e. g., finely ground talc or mica), Type X (on top and bottom), also Type Y (on bottom only).
2. Embedded in the top coating	*Moderately Coarse Mineral Matter* (e. g., coarsely ground talc) Type Y (on top only).
3. Embedded in the bottom coating	*Coarse Mineral Matter* (e. g., crushed slate, crushed brick or tile), Type Y (on top only).
4. Admixed with the top coating (Types X and Y)	May or may not be present. If present, consists of very fine mineral matter (e. g., clay, silica, limestone, slate flour, shale, etc.).
5. Admixed with the bottom coating (Types X and Y)	
6. Ash present in desaturated felt (i. e., ash on incineration).	

B—Bituminous Matter

1. Contained in the top coating.
2. Contained in the bottom coating.
3. Saturant of the felt.

C—Fibrous Matter

1. The desaturated felt.

The test strips for the separation of prepared roofing and shingles into their component parts shall be prepared as follows:

From the samples representing the average weight, a number of strips shall be cut, each measuring approximately 2-in. in width by

8 in. in length. These shall be weighed, the length and width measured to within 1 mm. or 1/32 in., and the weight calculated per 108 sq. ft. Any of the 2 by 8-in. strips whose weight varies more than 1.5 per cent from the average weight of the shipment, shall be rejected, and the remaining strips shall be retained for further examination.

NOTE. If a strip is cut to exactly 2 by 7.46 in. its weight in grams divided by two gives directly the weight in pounds per 108 sq. ft.

Detached Mineral Matter. (*A*-1). Remove the detached mineral particles from both sides of the 2-in. strips with a jet of

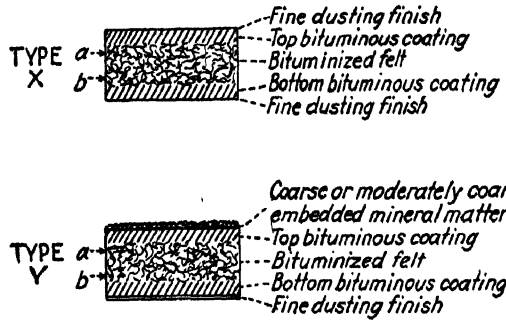
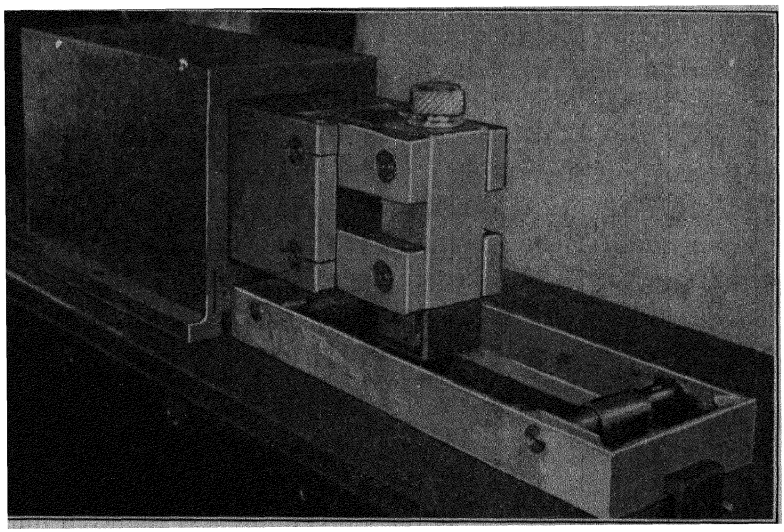


FIG. 311.—Types of Asphalt Roll-roofings and Shingles.

compressed air operating under a nozzle pressure of 25 lb. per sq. in. and reweigh in grams. Make a correction to include any loose mineral matter (see Test 65). Calculate the weight in pounds per 108 sq. ft. of the detached mineral matter (*A*-1) . . . . . (2)

Another means to remove the detached mineral matter [13] involves the use of a machine as illustrated in Fig. 312. This machine provides an abrasive or scrubbing action on the embedded granules by means of a standardized steel-wire brush actuated by a horizontally reciprocating crosshead driven by a motor with reduction gears. The brush is mounted on a floating head attached to the crosshead by ball-bearing toggles. The length of stroke of the brush is 6¾ in. and the load on the brush is approximately 4 lb. per sq. in. of its over-all area (1.25 sq. in.). The recommended test is 50 cycles (each of one forward and one backward stroke), weighing the specimens before and after the test to determine the granule loss. The machine is equipped with a starting device and counter which automatically stops the brush after 50 cycles.

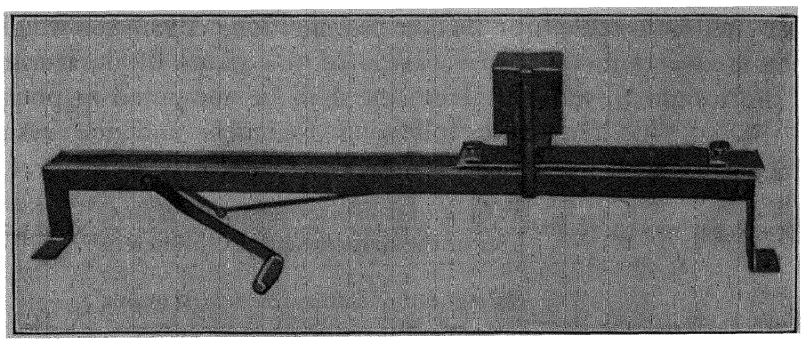
Figure 312 illustrates the complete machine, showing the floating head and brush lifted out of contact with the roofing test specimen. During an abrasion test the brush is dropped into contact



Courtesy A.S.T.M.

FIG. 312.—Rub-Test Machine, Type I.

with the specimen. The specimen holder is a cast aluminum-alloy pan, with built-in cam clamps, so that the specimen may, if desired, be kept immersed in water during the abrasion test. In making the



Courtesy A.S.T.M.

FIG. 313.—Rub-Test Machine, Type II.

"wet" test, it is preferable to collect, dry and weigh the granules removed, rather than to attempt to dry and weigh the specimen. A simpler type of machine is illustrated in Fig. 313. This machine

also employs a steel-wire brush, the length of stroke and pressure being approximately the same as in the preceding, but is operated by hand instead of by a motor, and requires counting of the number of strokes, since it has no automatic stopping device. It also differs in that the specimen moves instead of the brush. The test may either be run "dry," as shown in the illustration, or by using a pan, may be run "wet," with the specimen immersed in water during the test.

Moisture-free Felt (*C*–1). Eight of the 2 in. by 8-in. strips shall be extracted with benzol (boiling-point 80 to 82° C.), or c.p. carbon disulfide in a suitable extractor or centrifuge, and the extraction continued until the drippings have become colorless. The desaturated felt shall be removed from the extractor, being careful not to lose any adhering mineral matter, placed in a tared weighing bottle provided with a ground-glass stopper, heated in a ventilated oven at 225° F. (107° C.) ± 5° F. (3° C.) for thirty minutes, the stopper inserted, then cooled and weighed. The heating shall be repeated until the weight of the felt remains constant, as determined by two consecutive weighings taken not less than ten minutes apart, which shall show a further loss of not more than 0.1 per cent. Any adhering mineral matter shall be brushed off the felt, weighed and retained for further examination. The weight of said adhering mineral matter shall be deducted to obtain the weight of the moisture-free felt. The extract and accompanying mineral matter shall be retained for further examination. From the weights so obtained and the respective areas of the strips of desaturated felt, the weight of moisture-free felt shall be computed in pounds per 108 sq. ft., from the following formulas and the results averaged: ..... (3)

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of the strips in square centimeters}} \times 221.2$$

or

$$\text{Pounds per 108 sq. ft.} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of the strips in square inches}} \times 34.3$$

The extracted felt shall be retained for examination as to weight, thickness, strength, etc., in accordance with the tests described under Section E.

Ash in Moisture-free Felt (*A*–6). The strips of desaturated

felt obtained as described shall be cut into squares about 2 cm. on each side and thoroughly mixed. Pieces amounting to about 10 g. shall be selected at random and dried at 225° F. (107° C.) for thirty minutes. They shall be cooled in a desiccator, accurately weighed and then incinerated in a weighed crucible either over an open flame or in a muffle until all the carbon has been consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, cooled and reweighed. The percentage of ash shall be calculated on the basis of the moisture-free felt. . . (4)

Bituminous Saturation (B–3) in Moisture-free Felt. From the 2 by 8-in. strips, cut 40 strips about ¾ by 2 in. and weigh, this being a convenient size to handle. These strips shall then be separated into three horizontal sections. The small arrows *a* and *b*, to the left of the various types of roofing illustrated in Fig. 311, indicate approximately where the layers should be separated. The top bituminous coating, with the embedded mineral matter, shall be removed first.

NOTE. The purpose shall be to remove the entire coatings with some of the saturated felt adhering and no coatings left on the central section of saturated felt. The operator may accomplish this in any way that is most convenient. Care shall be taken to preserve the mineral surfacing with the layers to which it belongs if it becomes detached during the operation. A convenient manner to effect the separation consists in warming the strips in an air-oven at a temperature of approximately 176° F. (80° C.); then with a knife, the front and back coatings, respectively, shall be peeled off, care being taken to remove as little as possible of the saturated felt, and at the same time to make sure that all of the coatings and surfacings are stripped from the felt.

These three horizontal sections thus obtained shall be weighed separately and the total weight should agree within 1 per cent with the original weight of the strips which have been separated. The saturated felt (middle section) shall be extracted with benzol (boiling-point 80 to 82° C.) or c.p. carbon disulfide, in a suitable extractor or centrifuge and the extraction continued until the washings have become colorless. The desaturated felt shall be removed from the extractor, placed in a tared weighing bottle provided with a ground-glass stopper, heated in a ventilated oven at 225° F. ± 5° F. (107° C. ±3° C.) for 30 minutes, the stopper inserted, then cooled and weighed. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken with an intervening heating period of not less than 10 minutes, which shall show a further loss in weight of not more

than 0.1 per cent. Calculate the weight of bituminous saturation by difference. The saturant may be recovered by combining the extract and washings in a 200-ml. distilling flask, provided with a dropping funnel to add the solution during distillation, to which is attached a water-cooled condenser and a receiver, the latter having a vented connection to a vacuum pump. The distilling bulb is immersed in a bath having a temperature of about 212° F. (100° C.) if the solvent were carbon disulfide and a temperature of about 275° F. (135° C.) if benzol or chloroform were used as the solvent. Distillation shall be continued at this temperature at atmospheric pressure until no more solvent is evolved, after which vacuum shall be applied very gradually until a vacuum of 0.5 in. mercury pressure is attained. Great care shall be exercised in applying the vacuum gradually to prevent foaming. This vacuum shall be continued for one hour after the specified vacuum has been reached. The distilling flask shall then be disconnected and the residue represents the felt saturant and shall be weighed.

NOTE. Use the residue of bituminous saturation recovered from the felt for examining its physical and chemical characteristics.

Calculate the per cent of bituminous saturation (B–3) carried by the dry felt.....(5)

Calculate the weight in pounds per 108 sq. ft. of bituminous saturation (B–3) present in the dry felt, i.e., (5) × (3).....(6)

Bituminous Matter in the Top and Bottom Coatings Respectively (B–1 and B–2). Take six of the 2 in. by 8-in. strips, from which the detached mineral matter has been removed, and split them lengthwise by tearing the felt midway between the points *a* and *b*. Weigh and extract the top and bottom sections, respectively, in a suitable extractor or centrifuge. Desiccate and weigh the dry felt in each section. Separate the total embedded and admixed mineral matter recovered from each section, dry at 225° F. (170° C.), cool in a desiccator and weigh. Calculate the weight of bituminous saturant present, i.e., weight of felt recovered on extraction × (5). From the original weight of the top and bottom sections, respectively, subtract the combined weights of dry felt, bituminous saturant, embedded and admixed mineral matter. The difference represents the weight of bituminous matter in the surface-coating carried by that particular section.

Calculate the weight in pounds per 108 sq. ft. of bituminous matter (B–1) in the top coating.....(7 *a*)

Calculate the weight in pounds per 108 sq. ft. of bituminous matter (B–2) in the bottom coating.....(7 *b*)

Calculate the weight in pounds per 108 sq. ft. of total mineral matter embedded and admixed with the top coating.....(8 *a*)

Calculate the weight in pounds per 108 sq. ft. of total mineral matter embedded and admixed with the bottom coating.....(8 *b*)

To separate the bituminous constituents in the coatings for further examination and also to check the percentage of mineral matter, both detached and embedded in the top and bottom coatings, with that determined above, the detached mineral matter shall be brushed off from six weighed 2-in. by 8-in. strips. The strips shall again be weighed and the percentage calculated based on the original weight of strips. The outer portion of the top and bottom surface coatings, respectively, shall then be scraped off after the strips have been heated in an oven at 212° F. (100° C.) for 30 minutes. The coatings shall be scraped off by holding a dull knife at right angles to the strip of roofing supported on a firm level surface and drawing the blade sideways under moderate pressure. Care shall be taken to avoid scraping entirely through the surface coating. This is important. The scrapings shall then be weighed and dissolved in benzol (boiling-point 80 to 82° C.) or c.p. carbon disulfide; and the mineral matter separated by filtering or centrifuging and washing with successive portions of solvent. The mineral matter shall then be dried at 225° F. (107° C.), cooled in a desiccator and weighed. Calculate the percentage of mineral matter based on the original weight of roofing strips taken, which should check with that obtained previously. The bituminous matter in the scrapings shall be calculated by difference and reduced to a percentage basis on the original roofing strips used. The bituminous coating shall be recovered by evaporating off the solvent as described. Both surface coatings should be treated separately in this manner.

NOTE. Use the separated bituminous coatings for examining their physical and chemical characteristics.

Coarse or Moderately Coarse Embedded Mineral Matter (A–2), also Fine Admixed Mineral Matter (A–4) on the Top Coating of Type Y. The mineral matter recovered from the top sections of Type Y shall be boiled with 100 ml. water and washed through a 65-mesh copper wire screen with sufficient water to remove the fine mineral matter. The total aqueous suspension of mineral matter which passes through the screen shall then be recovered by filtering the washings through a weighed Gooch crucible, then dried at 225° F. (107° C.) and reweighed. This represents the fine mineral matter admixed with the top coating. The mineral constituents retained on the 65-mesh screen represent the coarse or moderately coarse mineral matter embedded in the top coating. This shall likewise be dried and weighed.

Calculate the weight in pounds per 108 sq. ft. of coarse or moderately coarse mineral matter (A–2) embedded in the top coating of Type Y.....(9)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter (A–4) admixed with the top coating of Type Y (*)..(10)

Fine Embedded and Admixed Mineral Matter (A–2 and A–4) in the Top Coating of Type X; Likewise Fine Embedded and Admixed Mineral Matter (A–3 and A–4) in the Bottom Coating of Types X and Y. Take five of the 2-in. by 8-in. strips, from which the detached mineral matter has been washed off with water, and scrape off the *outer* layer of the top and bottom coatings, respectively, by means of moderately rough sandpaper or a knife blade. Enough of the surface shall be scraped to remove every vestige of the fine embedded mineral matter, and at the same time care should be taken not to cut completely through the surface coatings into the saturated felt underneath. Then scrape off about 5.0 g. of the top and bottom surface coatings, respectively, with a sharp knife as described, taking care not to scrape entirely through the surface coatings. Ignite the 5.0-g. portions separately in a tared crucible until all the carbon has been consumed, add a few drops of ammonium carbonate solution, ignite to a dull red heat and weigh.

Calculate the *percentage* by weight of fine mineral matter admixed with the top coating (Type X).....(11 *a*)

* Up to 2 per cent admixed mineral matter probably adventitious and not filler.

Calculate the *percentage* by weight of fine mineral matter admixed with the bottom coating (Types X and Y) ..... (11 *b*)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter admixed with the top coating (Type X), i.e., (7*a*) × (11*a*) ..... (12 *a*)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter admixed with the bottom coating (Types X and Y), i.e., (7*b*) × (11*b*) ..... (12 *b*)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter embedded in the top coating (Type X), i.e., (8*a*) — (12*a*) ..... (13 *a*)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter embedded in the bottom coating (Types X and Y), i.e., (8*b*) — (12*b*) ..... (13 *b*)

Compilation of Results. The foregoing items shall be reported in pounds per 108 sq. ft. as shown in Table CXIV. If desired, the results may also be expressed in per cent by weight.

(C) RECOVERY AND EXAMINATION OF EXTRACTED COATINGS AND SATURATION

The bituminous constituents separated in Test 76 by extraction shall be recovered as described in Test 21b, whereupon they may be examined further by appropriate tests given in Chapter XXXII.

(D) EXAMINATION OF THE SEPARATED MINERAL SURFACING AND ADMIXED MINERAL CONSTITUENTS

The mineral constituents separated in Test 76 may be examined further by Tests 62, 63 and 64.[14]

(E) EXAMINATION OF THE SEPARATED FABRIC

The fabric separated upon extraction in Test 76 may be subjected to the following tests:

Test 77. Weight pér Unit Area ("Number"). This is expressed arbitrarily by the trade in terms of the so-called "number".

TABLE CXIV

	Type X	Type Y
Mineral Matter:		
Fine mineral matter embedded in top coating	Item (13*a*)	.....
Coarse or moderately coarse mineral matter embedded in top coating	.....	Item (9)
Fine mineral matter embedded in bottom coating	Item (13*b*)	Item (13*b*)
Fine mineral matter admixed with top coating	Item (12*a*)	Item (10)
Fine mineral matter admixed with bottom coating	Item (12*b*)	Item (12*b*)
Bituminous Matter:		
Contained in top coating	Item (7*a*)	Item (7*a*)
Contained in bottom coating	Item (7*b*)	Item (7*b*)
Saturant of the felt	Item (6)	Item (6)
Fibrous Matter:		
Felt after desaturation	Item (3)	Item (3)
Net weight of roofing material	Sum	Sum
Check total	Item (1)	Item (1)
Detached mineral matter	Item (2)	Item (2)
Packing material, nails and cement	Weight	Weight
Gross weight per 108 sq. ft.	Sum	Sum
Per Cent Ash:		
From desaturated felt	Item (4)	Item (4)

(*A*) In the case of paper, the "number" usually corresponds to the weight in pounds of a ream, consisting either of 500 sheets, for light papers, or 480 sheets, for heavy papers, each sheet measuring 24 in. by 36 in. Other arbitrary methods are also sometimes used embodying a different number of sheets, or sheets of other sizes. The following formulae will be found useful in calculating the "number":

$$\text{"Number"}\ (500{:}24 \times 36) = \frac{\text{gms.}}{\text{sq. cms.}} \times 6150 = \frac{\text{gms.}}{\text{sq. ins.}} \times 953$$

$$\text{"Number"}\ (480{:}\ 24 \times 36) = \frac{\text{gms.}}{\text{sq. cms.}} \times 5903 = \frac{\text{gms.}}{\text{sq. ins.}} \times 914$$

In stating the "number," the percentage of moisture present in the paper should also be given.

Moisture Content of Paper. The following procedure has been proposed [15] for ascertaining the moisture content of paper intended for electrical insulation purposes:

A weighing bottle, evaporating dish, thermometer, constant-

temperature oven, chemical balance and desiccator are necessary for the test. The weighing bottle should be of convenient size, about 65 mm. (2.56 in.) in height and 45 mm. (1.77 in.) in diameter with a wide mouth provided with a ground-glass stopper. The chemical balance should be sensitive to 0.1 mg.

The moisture content figure is used to calculate the percentage of acidity, size, and ash of the paper to the bone-dry basis. It shall be determined on 2 to 5-g. samples.

If the moisture content of the paper as received at the purchaser's works is desired or at any other stated time, it shall be made on the sample taken at the definitely stated time by cutting small pieces of approximately 6.5 sq. cm. (1 sq. in.) in area from the required number of rolls and immediately placed into an air-tight container.

NOTE. Since paper adjusts itself, as regards moisture content, to the humidity of the surrounding atmosphere in a very short time (2 or 3 minutes), special care must be exercised to transfer rapidly the sample from the roll or bundle to the container and from the container to the weighing bottle.

The sample before drying shall be weighed in the tared bottle with the stopper in place. The bottle shall then be placed in the oven at 100 to 105° C. (212 to 221° F.), the stopper removed and laid alongside of the bottle and the contents transferred to the drying dish. After one hour, while still in the oven, the sample shall be replaced in the weighing bottle, and the bottle stoppered and transferred to the desiccator. The stopper should be removed while the bottle cools. When the specimen and the bottle have cooled to room temperature, the stopper shall be replaced and the bottle with its contents weighed. This process shall be repeated at intervals of one hour until the difference in weight between two successive weighings is not more than 0.2 per cent of the weight of the specimen.

NOTE. The weighing bottle or sample should not be touched with the fingers during this test.

The moisture content shall be expressed:

(*a*) as a percentage of the weight of the dry sample, and
(*b*) as a percentage of the weight of the undried sample.

Weight of Conditioned Paper. Samples shall be conditioned in air maintained at a relative humidity between 60 and 65 per cent as measured with a sling psychrometer or its equivalent. The tem-

perature of the air shall be maintained as constant as possible at some temperature between the limits of 20 and 30° C. (68 and 86° F.). The samples should remain in the conditioned air for not less than 4 hours prior to the tests and should be supported so as to allow a free circulation around each sample.

Each specimen while in the conditioned atmosphere shall be cut accurately to any convenient size and accurately weighed. Specimens of sufficient size to weigh at least from 3 to 5 g. (0.11 to 0.18 oz.) should be taken. If it is not possible to do the weighing in the conditioned atmosphere, the specimen shall be placed in a weighing bottle and tightly stoppered with a ground-glass stopper before being removed from the conditioned atmosphere. The weight shall be calculated to and expressed as grams per square meter.

NOTE. Grams per square meter times 0.00142 equals pounds per 1000 sq in., from which the weight in pounds per ream of any size can be calculated if desired.

The apparent density shall be calculated from the thickness results obtained from the conditioned specimens and the weight by means of the following formula:

$$\text{Apparent Density, g. per cu. cm.} = \frac{\text{Wt., g. per sq. m.}}{\text{Thickness, in.}} \times 0.0000394$$

(*B*) In the case of felt, the "number" represents the weight in pounds of a ream consisting of 480 sheets, each measuring 12 in. × 12 in., the moisture content of which is arbitrarily set at 3.5 per cent. The following formula may be used (corrected for any carbonaceous matter present, as ascertained in Test 75).

$$\text{"Number" } (480 : 12 \times 12) = \frac{\text{gms.}}{\text{sq. cms.}} \times 983 = \frac{\text{gms.}}{\text{sq. ins.}} \times 152$$

Uncorrected "Number of Felt." The "uncorrected number" represents the weight of the felt as received in pounds per 480 sq. ft. To ascertain the "uncorrected number," unwind the first ten convolutions of each roll sampled, and with a knife and straight-edge cut cleanly across the sheet at right angles to the sides. Remove a section measuring 24 in. in the direction of the roll's length. From this, with a knife and template, cut three specimens, each measuring a "square foot," one from the center, and one close to each edge of the sheet. A "square foot" shall imply 144 sq. in. within that

degree of accuracy which will not affect the weight thereof on a paper makers' scale more or less than ½ lb. per 480 sq. ft., from 1 sq. ft. area absolute. Rapidly weigh each square foot specimen and compute the "number" (i.e., the weight in pounds per 480 sq. ft.). Average the three readings for each roll sampled, and from these results, compute the minimum, maximum, and average "uncorrected number" of the felt. Unless the square foot samples are tested immediately by the laboratory, they should be preserved preparatory to testing, as soon as they are cut from the sheet, in waxed or waterproofed string-tied envelopes to prevent any change in moisture content.

Moisture Content of Felt. With the least possible delay after the rolls have been sampled, cut from each square foot specimen a 2-in. strip (within ½ in.) across the fiber grain. Weigh the strips in aggregate, and then heat for not less than one hour in an oven having a free circulation of air, the internal temperature of which is uniformly maintained between 215 and 225° F. Remove the felt from the oven and weigh quickly, to prevent reabsorption of moisture. Replace the specimens in the oven and reweigh at intervals of fifteen minutes, until no further loss is noted. Compute the average percentage by weight of moisture in the felt as received.

Another method [16] consists in distilling a weighed quantity of the felt with perchlorethylene ($CCl_2.CCl_2$), collecting the distillate in a graduated tube and measuring the volume of water which floats on the surface.

Corrected "Number of Felt." The "corrected number" represents the weight of the moisture-free felt in pounds per 480 sq. ft. The minimum, maximum and average "corrected number" of the felt is obtained by deducting the average percentage of moisture from the "uncorrected number."

(*C*) In the case of woven fabrics, the "number" represents the weight in ounces per lineal yard of a specified width, the moisture content being arbitrarily fixed at 6.5 per cent.[17] With burlaps the width is 40 in., with "regular" ducks 29 in., etc.

Test 78. Thickness. This shall be ascertained as described in Test 66.

Test 79. Strength. The strength is determined by one of the following methods:

(*A*) In the case of paper, the sample after being "conditioned" [18] may be tested for tensile breaking strength by the following method which has been standardized:

(*a*) A testing machine of the dead-weight pendulum type suitably designed for testing paper shall be used. The machine shall preferably be power driven.

(*b*) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(*c*) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over, and to 0.5 lb. or 0.5 kg. or less for testing paper breaking under 22.7 kg. (50 lb.).

From each of the samples selected, specimens shall be cut at least ten in the machine direction and if practicable, ten in the cross-machine direction. The specimens shall not exceed 2.54 cm. (1 in.) in width and shall be 25.4 cm. (10 in.) in length with clean-cut edges.

The ratio of the clearance distance between jaws to the width of the specimen shall not be less than 5 to 1 nor more than 10 to 1. The rate of travel of the movable jaw shall be constant. It shall preferably be 30.5 cm. (12 in.) per minute, but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per minute provided it is constant.

All readings obtained when the paper breaks at or in the jaws shall be rejected. The results of the machine-direction specimens and the cross-machine-direction specimens shall be reported separately. The results shall be reported in kilograms or pounds together with the width of the specimen in centimeters or inches and also the average thickness. The maximum, minimum, and average breaking load shall be reported for the machine direction and the cross-machine direction.

The bursting strength of the paper may be ascertained by the Mullen Tester (Fig. 307) or an analogous type of tester, the procedure having similarly been standardized as follows:

The testing machine shall have a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber shall be filled with glycerin or other suitable pressure medium and shall contain no air spaces. The test specimen shall be held in position over

the diaphragm in a clamp having a circular hole approximately 6.44 sq. cm. (1 sq. in.) in area so that the diaphragm will force the paper into the hole when pressure is applied under the diaphragm. The pressure exerted on the diaphragm shall be indicated on a dial, graduated to at least 0.23 kg. (0.5 lb.) for papers giving bursting strengths 18 kg. (40 lb.) or under. A dial graduated 0.45 kg. (1 lb.) may be used in testing papers which give higher bursting strengths. The machine may be either hand-operated or power-driven, the latter being preferable.

If practicable, the specimen shall be so cut from the stock as to permit ten bursting tests on a line across the sheet or roll. Ten bursts shall be made, five with one side of the paper uppermost and five with the other side uppermost. The testing machine shall be driven at a uniform speed of 120 r.p.m. until the specimen bursts. The report shall include the average, the maximum, and the minimum results obtained.

(*B*) In the case of felt, expose the desaturated fabric for three days to air at 77° F., completely saturated with moisture, and then find its tensile strength as in Test 67. The reason for this is because the strength of the dry felt is increased materially during the process of extracting with solvents, but it may again be brought to correspond closely with its original strength by treating as described.

The following figures will illustrate this point, viz.: strength original felt (before extraction), 23.3 lb. (average of 10 tests); original felt upon heating to 265° F. for five minutes, cooling in a desiccator and testing immediately, 26.9 lb.; original felt upon extracting with benzol in Soxhlet for five hours, cooling in desiccator and testing immediately, 36.4 lb.; extracted felt exposed three days to air at 77° F. carrying 30 per cent moisture, 33.6 lb.; extracted felt exposed three days to air at 77° F. completely saturated with moisture, 27.6 lb.

(*C*) In the case of woven fabrics (e.g., cotton cloth, duck, burlap, etc.), it is customary to find the tensile strength when the desiccated fabric has a moisture regain, which it assumes on exposure for at least four hours to an atmosphere having a relative humidity of 65 per cent at 70° F. (21.1° C.).

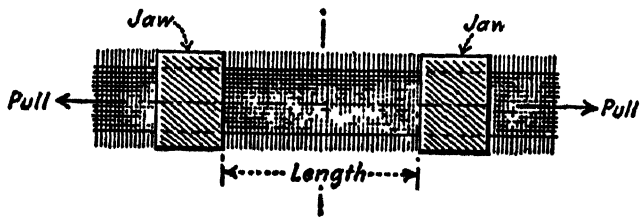
For the determination of tensile strength [19] a swatch of sufficient length as specified in Table CXV shall be cut the entire width

TABLE CXV

TENSILE STRENGTH TEST METHODS

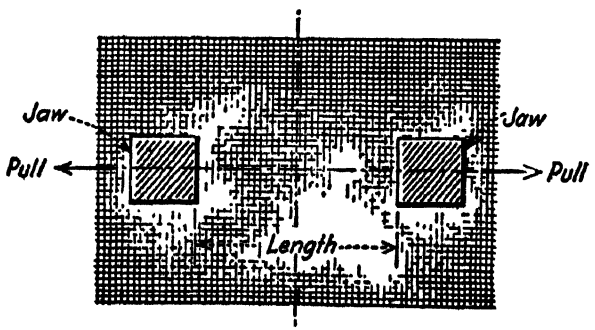
Method No.	No. 1 Strip Method	No. 2 Grab Method
Length of specimens, in.	7	6
Width of specimens, in.:		
If thread count is 80 or over	1⅛	4
If thread count is 50 to 80	1¼	
If thread count is less than 50	1½	
Ravelled to standard or specified threads per in.	1	..
Minimum width of bottom or back jaws, in.	1½	2
Width of top or front jaws, in.	1½ (min.)	1
Distance between jaws, in.	3	3
Speed of pulling jaw, in. per minute	12	12
Number of specimens each, warp and filling	5	5

of the cloth, and ten test specimens shall be cut from this swatch, five with the long dimension in the direction of the warp and five in the direction of the filling. The specimens shall be fairly repre-



Courtesy A.S.T.M.

FIG. 314.—Illustration of Strip Test.



Courtesy A.S.T.M.

FIG. 315.—Illustration of Grab Test.

sentative of the material. Except in narrow fabrics (less than 3 in. in width), all specimens shall be taken at least 1 in. from the selvage.

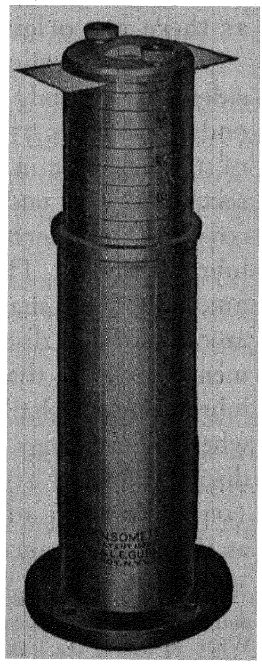
Strip Test (*Fig.* 314). Under this method a long narrow strip

of the fabric shall be clamped at each end by the jaws of the testing machine and strained to the point of rupture.

Grab Test (*Fig.* 315). Under this method the testing machine jaws, each of definite width, shall be made to reach into the body of a rectangular piece of fabric and shall be clamped a definite distance apart. Care shall be taken that the same set of threads are embraced by both pairs of jaws. The specimen shall then be strained to the point of rupture. The average of the five individual tests on the warp shall be the strength of the warp and the average of the five individual tests on the filling shall be the strength of the filling.

Test 80. Porosity. This test is applicable to paper or felt and measures the porosity as indicated by the "air resistance," by an instrument known as the "densometer," illustrated in Fig. 316. The procedure has been standardized as follows:[20]

(*a*) The instrument shall consist of two aluminium open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular aperture in the closed end and a flat ring clamp for holding the specimen across this aperture.



Courtesy W. & L. E. Gurley

FIG. 316.—The Densometer.

(*b*) The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.60 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) long, 3 mm. (0.12 in.) wide and approximately 1.5 mm. (0.06 in.) thick shall be mounted vertically and equidistantly on the inner surface of the fixed cylinder to act as guide-tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 ml. and shall have a total range of 350 ml. It shall be 25.4 cm. (10 in.) high and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts, 567 g. ± 0.5 g. (20 oz. ± 0.018 oz.). The movable

cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area. When this aperture is too large for the specimen a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

NOTE. Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

The specimens shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width cut from the original samples, and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) wide and at least 30 cm. (12 in.) long. There shall be as many specimens as there are original samples.

The fixed cylinder shall be placed on a rigid support so that its sides are vertical. A lubricating oil with viscosity of 60 to 70 seconds Saybolt at 37.8° C. (100° F.) shall be placed in it to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp on the movable cylinder completely covering the aperture. The movable cylinder is then floated on the lubricating oil. The time required for the displacement of a certain amount of air shall be noted with a stop-watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 seconds. The apparatus with its content of lubricating oil shall be at the temperature of the conditioning room when the readings are taken. The time in seconds required for the displacement of 100 ml. (6.1 cu. in.) of air through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the paper is known as the air resistance of the paper.

NOTE. The clamp shall be tested for leakage by substituting a piece of tinfoil 0.05 mm. (0.002 in.) in thickness for the paper and testing in the manner described above. When so tested, the leakage shall not exceed the rate of 50 ml. in five hours.

The proper procedure for clamping the specimen or tinfoil is to turn both knurled nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak.

Oil is used in preference to distilled water because it does not corrode aluminium whereas distilled water does.

Precautions should be taken to avoid subjecting the apparatus to vibration as this condition would increase the rate of air displacement.

The report shall include the following:

(*a*) the number of seconds required for the displacement of 100 ml. (6.1 cu. in.) of air;
(*b*) the area of paper through which the air was displaced;
(*c*) the thickness of the paper;
(*d*) the room temperature.

NOTE. The following values show the probable accuracy obtainable in the air-resistance tests:

Air Resistance	Accuracy
40 seconds	± 5 per cent
100 seconds	± 6 per cent
200 seconds	± 8 per cent
300 seconds	± 10 per cent

Test 81. Speed with which the Felt Will Saturate. This test is designed to ascertain the relative speeds with which roofing felt will saturate with bituminous substances at operating temperatures. Two alternate procedures are recommended for the purpose:

(*a*) *Vertical Method.*[21] Strips of moisture-free felt 2.5 cm. wide cut lengthwise from the sheet (the length being immaterial) are marked with a pencil 1 cm. and 4 cm. from one end. They are then supported vertically and immersed up to the 1-cm. mark in xylol distilling between 135 and 145° C. by the standard Engler distillation method, which is maintained at 77° F. By means of a stop-watch the time is recorded during which the xylol rises by capillarity to the 4-cm. mark (i.e., exactly 3 cm. above the level of the xylol). The number of seconds is an index of the speed with which the felt will saturate.

(*b*) *Horizontal Method.*[22] Samples measuring 2 by 2 in. are gently floated on the surface of a mixture of clear paraffin oil with a clear light engine oil, having a Saybolt viscosity of 253 seconds at 72° F. and 125 seconds at 100° F. One specimen is floated right side up, and another upside down on the oil maintained at 72° F. When the uppermost surface is judged to be darkened by the oil to an extent of 90 per cent of its area, the time in seconds is recorded. The number of seconds for the oil to penetrate from each side of the sheet is termed the "oil penetration" for the respective side of the felt. To facilitate making the test, it is recommended that a series of oil mixtures be prepared, each having the prescribed Saybolt viscosity at 60°, 65°, 70°, etc., up to 85° F. respectively, so that

the particular oil may be used, depending upon the room temperature, thus avoiding the necessity of bringing the oil to the proper temperature. The moisture content of the felt affects the results. It has also been suggested that the weight of oil absorbed be ascertained and expressed in per cent by weight of the dry felt.

Test 82. Saturating Capacity. This test is intended to serve as an indication of the quantity of saturant which the felt may be expected to absorb under definite machine conditions. It consists in saturating a sample of the moisture-free felt with a measured volume of kerosene, and hence has been termed the "kerosene test." [28] It is performed as follows: A representative sample of the felt measuring exactly 2 by 5 in. is placed in an open tared weighing bottle and thoroughly dried for at least one hour in an air-oven of which the internal temperature is uniformly maintained at 220–225° F. It is then cooled in a desiccator, whereupon the cap of the bottle is inserted, and weighed together. A 25-ml. burette graduated in 0.1 ml. is filled with kerosene at room temperature, which is allowed to flow in a thin stream against the surface of the felt supported horizontally on the upper rim of a small beaker. The beaker may be conveniently moved about to distribute the kerosene over the surface of the felt until it becomes uniform in color. The stream of kerosene is then retarded, so that it will flow drop by drop, until a point is reached when the surface of the felt assumes a characteristic glazed appearance when viewed at an angle. The end-point is quite sharp. A corner of the felt is then lightly grasped with a pair of tongs and held vertically over the beaker for exactly thirty seconds, whereupon the edges are rinsed lightly against the rim of the beaker to catch any drippings that may have accumulated. Any kerosene collected in the beaker is poured back into the burette before the final reading is taken.

If c represents the number of ml. kerosene required to saturate the specimen.

w_1 the weight in grams of the 2 by 5-in. specimen of moisture-free felt.

g_1 the specific gravity of the saturant at 77° F. (which for convenience may be assumed to be 1.04 in the case of asphaltic saturants).

s the maximum percentage by weight of saturation theoreti-

cally possible, based upon the weight of the moisture-free felt.

Then $$s = \frac{100\, c g_1}{w_1}$$

An alternate procedure consists in immersing the weighed specimen of moisture-free felt in kerosene at 77° F. and subjecting it to a vacuum of at least 28 in. for 3 minutes, whereupon it is allowed to remain in the kerosene for another 2 minutes at atmospheric pressure. The felt is then removed, allowed to drain 2 minutes while held diagonally in a vertical position, then placed in a tared weighing bottle, and reweighed.

If w_2 represents the weight of felt and kerosene combined, and g_2 the specific gravity of the kerosene at 77° F.

Then $$s = 100 \frac{g_1}{g_2}\left(\frac{w_2}{w_1} - 1\right)$$

Test 83. Fiber Composition. The following method has been proposed.[24] A sample of 25 g. shall be obtained by cutting at least 100 small sections from different portions of the various samples of felt under examination.

Microscope. The microscope shall be capable of giving a magnification of not less than 100 diameters and shall be of the compound type, provided with a mechanical stage, and substage condenser.

Graticule. The graticule or cross-line disk, for insertion into the eyepiece of the microscope, shall be ruled with two fine lines intersecting approximately at right angles.

Dropper. The dropper shall consist of a glass tube 20 cm. in length and 6 mm. in internal diameter fitted at one end with a rubber bulb and having the other end carefully smoothed but not constricted, in a flame.

Herzberg Stain. The Wilhelm Herzberg stain used shall be prepared as follows:

Solution A. Dissolve 50 g. of dry zinc chloride (fused sticks) in 25 ml. of distilled water, added to the zinc chloride bottle with a pipette. Stopper the bottle and shake well. There should be approximately 40 ml. of the solution.

The specific gravity of the solution determined at 28° C. shall be 1.8. If the specific gravity is not 1.8, add distilled water until the specific gravity is 1.8, and then pour the solution into a tall cylinder.

Solution B. Wash the thermometer, hydrometer, and the original bottle containing the zinc chloride with a portion of 12.5 ml. of distilled water. Add this to Solution *A* in the tall cylinder.

Dissolve 5.25 g. of potassium iodide and 0.25 g. of iodine in the balance of the 12.5 ml. of distilled water, and add to Solution *A* in the tall cylinder.

Stir the solution in the tall cylinder well, and place in the dark. The following day, pipette off the clear portion into a black bottle, leaving 3 or 4 ml. of the solution above the sediment. Add a leaf of crystal iodine.

This stain should ordinarily be found to be satisfactory for at least two weeks. If for any reason the stain is unsatisfactory, it shall not be adjusted but new stain shall be prepared. On referee tests, no stain over seven days old shall be used.

Ten grams of the sample shall be treated in a 400-ml. beaker with 200 ml. of water and heated to boiling. Stir the liquid thoroughly, preferably with a mechanical stirrer, until the sample is well pulped and the fibers well separated. The mass shall then be placed on a No. 100 (149 micron) sieve and washed thoroughly with water. The moist mass of paper retained on the sieve shall then be rolled into a ball and worked between the fingers to loosen the fibers thoroughly. Approximately one-fourth of this sample shall then be placed in a stoppered bottle and shaken thoroughly with 250 ml. of water until the fiber mass is thoroughly disintegrated and separated. Transfer three 25-ml. portions of this suspension to separate bottles and dilute each with water to 250 ml., so as to obtain suspensions of about 0.1 per cent pulp. These suspensions shall be thoroughly shaken before withdrawing a sample for the microscope test described below.

Procedure: The fibers shall be transferred from the 0.1 per cent pulp suspension to the microscopic slide as follows: Thoroughly mix the fibers and water by shaking thoroughly, quickly insert the dropper into the mixture 5 cm. (2 in.) below the surface, expel two bubbles of air from the dropper, then fill the tube to a distance of

about 13 mm. (½ in.). Transfer the contents of the dropper to the slide, making four drops, completely emptying it. Repeat this procedure until the slide is uniformly covered with drops of the suspension, then place the slide in an air-bath until dry. Add stain to the slide by means of an ordinary dropper so as to uniformly cover the fibers with the stain. Allow to stand for two minutes, remove most of the excess stain with hard, clean-cut filter paper and press down a second slide or suitable cover-glasses on the fibers. Remove the remaining excess of stain with absorbent paper.

Place the round-glass graticule on the diaphragm of the microscope. The point formed by the intersection of the two fine lines ruled on the graticule shall be used as the eyepiece reference point. Starting at one side of the slide about one-fourth of the way from the top, the slide shall be moved in a straight line across its entire width, by means of the mechanical stage. In moving thus through the field of view, each fiber or part of a fiber shall be counted which touches the eyepiece reference-point. Some of the fibers may be long and come under the point twice or more, but they shall be counted each time. If aggregations of fibers, such as occur in ground wood, are encountered, the number of single fibers in the aggregation shall be estimated and figured as if the individual fibers were completely separated. Another path across the slide shall be selected, about half way down from the top. The entire counting process shall be repeated and the results added to those obtained above. Repeat again over a path about three-fourths of the way down from the top and add the results to the total as before. Select two or more up-and-down paths and repeat the process. In this manner not less than 300 fibers shall be counted on each slide, using one or more up-and-down paths if necessary to obtain the required number. Each slide shall be calculated separately and the results for the different constituents compared with the average result. These shall agree with the average within the following permissible variations:

Cotton	$\pm \sqrt{\text{Average}}$
Chemical wood	$\pm (\sqrt{\text{Average}} + 1)$
Mechanical wood	$\pm (\sqrt{\text{Average}} + 2)$
Jute and manila	$\pm (\sqrt{\text{Average}} + 3)$

If such agreement is not obtained, additional slides shall be made from new 0.1 per cent suspensions and examined until three results are obtained which fall within the specified limits. The average of three such slides shall be reported as the result of the analysis.

NOTE. To illustrate the application of the above tolerances, suppose the average of three slides for a given constituent is found to be 33 per cent. The square root of 33 is 5.7. Therefore, all individual results shall lie between 27.3 and 38.7 per cent, if the constituent is cotton, and between 26.3 and 39.7 per cent if chemical wood, etc.

The fibers may be recognized (Note 3) by the colors developed by the stain, and by the surface appearance and markings on the fibers themselves.

Color	Fiber Composition
Red	Linen, cotton, bleached Manila hemp.
Blue	Chemically prepared fibers from wood, straw, and esparto.
Yellow (various shades to brown)	Ground wood, sawdust, jute, and Manila (Note 1).
Colorless	Wool (Note 2).

NOTES

Note 1. Ground wood and sawdust (mechanical wood) may be distinguished from jute and manila by the broken ends of the fibers, round, circular pits on the surface of the fibers as well as ray-like structure of portions of certain of the woods.

Note 2. Wool fibers are also often variously colored from the dye to which the wool has been subjected prior to its use as a raw material for roofing felts and in such cases must be recognized by its regular cylindrical appearance, scale, and hook-like markings.

Note 3. Proficience and experience in recognizing the appearance and structure of the various fibers are essential for accurate testing. It is recommended that all operators carrying on the tests obtain *Technologic Paper No. 250*, of the Bureau of Standards, entitled "Pulp and Paper Fiber Composition Standards," which contains color plates of various fibers treated with the Herzberg stain. A study of these plates, together with preparation of slides from known materials, is necessary to familiarize the operator with the characteristics of the various fibers and thereby perfect his technique.

The proportion of fibers found shall be reported in terms of percentage by counts of the total fiber composition and the following classes reported:

1. Cotton, linen, and bleached Manila hemp..... ——
2. Chemical wood, straw, and esparto..... ——
3. Mechanical wood..... ——
4. Jute and Manila..... ——
5. Wool..... ——
6. Unidentified fibers..... ——

In the foregoing test the following types of fibers will be differentiated:

(*a*) Fibers occurring naturally in an unlignified state are stained red (e.g., linen, cotton, hemp, ramie and parts of manila and esparto).

(*b*) Fibers occurring naturally in a lignified state, but which upon treatment with chemicals are freed from their lignin content are stained blue (e.g., wood, straw, esparto, jute, manila and other cellulose fibers).

(*c*) Lignified fibers (e.g., ground wood, sawdust, untreated straw, jute, manila, turf, etc.) as well as undyed silk fibers are stained yellowish to brown.

The following procedure may be used to distinguish wool fibers when they are dyed in various colors and mixed with cellulose fibers.[25]

After placing the fibers on a slide glass, cover with a few drops of a 30 per cent solution of caustic soda and heat gently over a flame until boiling just takes place. Remove immediately, and examine under the microscope. Wool fibers appear very much swollen and full of bubbles, whereas cellulose fibers remain unchanged.

Wool fibers may be determined quantitatively[26] by treating the felt with 80 per cent sulfuric acid for 3 hours, whereupon the wool fibers remain as residue. A correction must be made if the felt contains mechanical wood fibers.

To distinguish between bleached sulfate and bleached sulfite (i.e., kraft) fibers, the following method has been proposed:[27] One gram of sodium carbonate is dissolved in 175 ml. of distilled water, and to this solution 1 g. of C.P. brazilin * is added, stirring until dissolved. The solution gives sharper differences when used fresh, but if retained for future use, air should be excluded. The solution may be applied directly to the sample to be identified, the excess being removed with hardened filter paper, a few drops of U.S.P. white paraffin oil being placed on the slide, and any excess oil being removed. Bleached sulfite is stained a wine-red color, and bleached sulfate (kraft) is stained a purple color by the brazilin solution.

Woven fabrics are usually tested in the following manner: From a piece of the desaturated fabric (from which any sizing has been removed by bringing to a boil in a 0.5 per cent sodium hydroxide solution), take at random 5 threads in the direction of the warp

* MacAndrews & Forbes, New York City.

and 5 in the direction of the filling and separate the fibers in water on a microscope slide, so that the fibers are uniformly distributed. Dry the slides so prepared until all the water is removed, cool and add a drop of the Herzberg solution. Cover the stained fibers with a cover-glass, draw off the excess stain, and examine under a microscope with a magnification of about 100 diameters.

CHAPTER XXXV

EXAMINATION OF BITUMINOUS-SOLVENT COMPOSITIONS

Bituminous lacquers, cements, varnishes, enamels and japans are all characterized by the presence of a volatile solvent with a bituminous base, combined in the form of "vehicle." Depending upon whether or not the bituminous-solvent compositions contain a pigment or filler, they may be divided into two general classes, viz.:

(1) Pigment or filler absent: including bituminous varnishes and japans, also certain bituminous lacquers and cements.
(2) Pigment or filler present: including bituminous enamels, also certain bituminous lacquers and cements.

The first class consists of a vehicle made up of a solvent and base. The second consists of a pigment or filler combined with a vehicle, the latter similarly being made up of a solvent and base. The bituminous base may be composed of bituminous matter, with or without the presence of animal and vegetable oils or fats, resins or metallic dryers. In making an analysis of the lacquer, cement, varnish, enamel or japan, the following components are separated and examined, viz.: (1) solvent, (2) pigment or filler, (3) base.[1]

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT

Test 84. Résumé of Physical Tests. The following constitutes a brief description of the more important physical tests recommended for bituminous-solvent compositions.

(*a*) *Specific Gravity.* In the case of fluid materials, this shall be ascertained by means of a hydrometer (Test 7*a*) or a Westphal balance (Test 7*b*). Plastic cements shall be tested by means of a pycnometer (Test 7*d*). In either case, care should be taken to prevent evaporation of the solvent. The weight per gallon may be calculated from the specific gravity, by multiplying by 100 and dividing the product by 12.

(*b*) *Viscosity.* This is of value for purposes of factory control. If the material is sufficiently fluid, the Engler method (Test 8*a*) may be used, otherwise special viscosimeters may be used for the purpose, including the following:

Air-bubble Types, such as the Gardner-Holt and Collins types.
Falling-weight Types, such as the A.S.T.M. viscosity tester.[2]
Torsion Types, including the Stormer and MacMichael viscosimeters.
Efflux Types, including the General Electric cup, the Parlin viscosity cup, etc.
A description of these will be found elsewhere.[3]

(*c*) *Plasticity and Mobility.* Various instruments have been described for testing these properties[3] including the Bingham-Green Plastometer, the Gardner-Parks Mobilometer, the P.P.G. consistency tester, the Vacuum Plastometer (Gregory, Rassweiler and Lampert) the Hickson Penetration method, and the Gardner Flowmeter.

(*d*) *Flash-point.* This is of value in controlling the safety requirements. The Pensky-Martens Closed Tester (Test 17*a*) or the Tag Closed Tester (Test 17*c*) may be used for the purpose.

(*e*) *Spreading Capacity and Workability.* This test is of interest in determining the ease with which the material is applied and the surface area covered under normal working conditions. The material is spread on a clean surface of a character on which it is intended to be used, by skilled workmen operating under normal conditions of temperature, light and humidity. The workability of the material is carefully noted and the area covered per gallon recorded.

(*f*) *Draining Test.* This test has also been termed the "working viscosity test" and records the behavior of varnishes, japans and enamels when used for dipping purposes. It has been standardized as follows:[4]

A strip of sheet copper or brass about 4 cm. (1.57 in.) wide, 35 cm. (13.78 in.) long, and 0.127 mm. (0.005 in.) thick, shall be immersed in the varnish at a room temperature of approximately 20° C. (58° F.) up to a line previously drawn across the strip 1 in. from the top. The sheet shall be withdrawn at a slow and uniform rate (about 38 cm.-15 in. per minute), care being taken that the

varnish is free from air bubbles. The specimen shall be permitted to drain thoroughly at room temperature while suspended in a vertical position. It shall then be dried or baked (according to the type of the varnish) until dry.

The thickness of the specimen in mils shall be measured at points 5.1 cm. (2 in.), 17.8 cm. (7 in.) and 30.5 cm. (12 in.), respectively, from the line to which the specimen was immersed. The thickness of each film in mils at the three points specified above shall be recorded. The difference between the thickness at the upper point (5.1 cm.) and that at the lower point (30.5 cm.) shall be taken as a measure of the variation in the film thickness caused by draining.

(*g*) *Time of Drying.* The times which elapse until the coating sets to the touch (i.e., ceases to be tacky), also when it sets to a firm, tough coating, furnish an indication of the speed in which the composition dries. This test has been standardized as follows:[4] Specimens for this test shall be pieces of thoroughly cleaned, smooth sheet copper or brass about 4 cm. (1.57 in.) wide and 20 cm. (7.88 in.) long and about 0.127 mm. (0.005 in.) thick.

The specimen shall be dipped once in the varnish at a room temperature of approximately 20° C. (68° F.) and withdrawn slowly and uniformly (about 38 cm.-15 in.-per minute). The consistency of the varnish shall be first so adjusted by trial that, when dry, the average thickness of the film of varnish on each side of the metal shall be between 0.022 and 0.026 mm. (0.0009 and 0.001 in.). Care shall be taken before dipping the specimens that the varnish has stood in the dipping tank for a sufficient length of time to be free from air bubbles.

Specimens of air-drying varnish shall be dried in dust-free air at a room temperature of approximately 20° C. (68° F.). In the case of baking varnishes, six specimens shall be dipped and allowed to drain at a room temperature of approximately 20° C. (68° F.) until the varnish is set as indicated when the impression left on the surface by pressing lightly thereon with a finger at a point approximately 2 in. from the bottom will not become obliterated by further flow of the material. They are then to be dried in dust-free air in an oven at 105 to 110° C. (221 to 230° F.). At the end of the first 30 minutes, and again at the end of each 10-minute period

thereafter, one specimen shall be taken from the oven and examined. In the case of slow-drying varnishes, this 10-minute period may be lengthened at the discretion of the operator.

NOTE. The size of oven and the amount of ventilation have a considerable effect on the drying time of varnishes. For this reason the analytical type of electrically-heated oven should be used.

The varnish shall be considered dry when a piece of kraft paper 38 mm. (1.49 in.) in width and 152 mm. (6 in.) in length and approximately 0.063 mm. (0.0025 in.) in thickness does not adhere to the varnish when it is pressed on the surface of the varnish for one minute by a cylindrical 1-lb. weight, 1 in. in diameter. The kraft paper shall be applied in the vicinity of the center of the specimen and at right angles to it. The test shall be made at a temperature of approximately 20° C. (68° F.).

NOTE. Certain types of varnishes dry with what is commonly known as a "tack"; but these types are rare. However, when testing them for drying time, it will be found that the kraft paper adheres to the varnish film and offers a certain amount of resistance to removal. On continued drying, the resistance to removal reaches constancy and is not changed by further drying. Therefore, the drying time is reported as the number of hours required to first reach constancy and the varnish should be reported as drying with a "tack."

(*h*) *Hiding Power.* This is the property to obliterate any background upon which the composition may be spread under accurately regulated conditions. The methods usually employed include the use of the Pfund Crytometer,[5] Hallett's Hiding Meter,[6] the Bruce apparatus,[7] and others.[8]

(*i*) *Color.* Various instruments have been described[9] for measuring the color of dried films, including the Visual Spectrophotometer, the G.E. Recording Color Analyzer, the Razek-Mulder Color Analyzer, the A.P.C. Spectrophotometer, the Appel-Hickson Spectrophotometer, the Munsell Universal Photometer, the Hess-Ives Tint Photometer, the Westinghouse Photo-Electric Color Matcher, the Watson Color Analyzer, etc.

(*j*) *Gloss.* Similarly, various methods have been proposed for measuring the gloss of dried films, including the Hunter Glossmeter, the Pfund Glossimeter, the Ingersoll Glarimeter,[10] the Sward-Levy Gloss Comparator, the Hunter Gloss Comparator, etc.[8]

(*k*) *Hardness, Abrasion and Adhesion.* Numerous devices

have been proposed for ascertaining these properties, a description of which will be found elsewhere.[3]

(*l*) *Water Absorption.* A specimen is spread on glass as in (*e*) and when dry, immersed in water at room temperature (approximately 70° F.) for twenty-four hours. The film is then examined for adhesion, toughness, and in the case of insulating varnishes the dielectric strength is determined as in (*o*).

(*m*) *Resistance to Heat.* This is of special value in the case of baking japans and insulating varnishes. This test has been standardized as follows:[4]

Specimens shall be prepared by dipping pieces of thoroughly cleaned, smooth sheet copper or sheet brass about 20 cm. (7.88 in.) in length, 3 cm. (1.18 in.) in width and 0.127 mm. (0.005 in.) in thickness, into the varnish. The consistency of the varnish shall previously be adjusted by trial so that, when dry, the average thickness of the film of varnish on each side of the metal shall be between 0.022 and 0.026 mm. (0.0009 and 0.001 in.).[11]

Each specimen shall be dipped in the varnish at room temperature of approximately 20° C. (68° F.) and withdrawn slowly and uniformly (about 38 cm.-15 in.-per minute). The varnish shall be allowed to dry in the air (if an air-drying varnish) or baked in an oven at 105 to 110° C. (221 to 230° F.) (if a baking varnish) until dry. As soon as the first coat of varnish is dry, and the specimen is at room temperature, it shall be dipped again in the varnish in the reverse direction so as to give a uniform thickness of coating. The second coat of varnish shall be allowed to dry in the same manner as the first coat. When the second coat of varnish is dry, the specimen shall be measured. The thickness of varnish on each side of the metal shall be between 0.044 and 0.052 mm. (0.00173 and 0.00205 in.).

The specimens shall be placed in a uniformly heated oven, in which the temperature is maintained at 105 to 110° C. (221 to 230° F.). A specimen shall be removed at the end of the first 24 hours and every 24 hours thereafter.

NOTE. Because of temperature variations between different levels in the oven, the specimen should be placed in the oven with the 8-in. dimension horizontal and the 1-in. dimension vertical. All specimens should be at the same level in the oven.

Each specimen, after heating, shall be tested at room tempera-

ture of approximately 20° C. (68° F.) by bending it through 180 deg. around a rod 0.32 cm. (0.125 in.) in diameter. The number of hours of baking of the specimen which first showed cracking of the varnish film shall be reported, together with the make and type number of the oven used.

NOTE 1. The size of oven and the amount of ventilation have a considerable effect on the heat endurance of varnish films. For this reason the analytical type of electrically-heated oven should be used.

NOTE 2. The temperature of the oven should be held as closely as possible to the mean (107.5° C., 225.5° F.). A difference of 5° C. (9° F.), in this temperature range, when continuously maintained, has a very considerable effect (approximately 25 per cent) on the life test of a varnish.

NOTE 3. As the "grain" of the copper influences the result of the test, the heat endurance test specimens should be prepared by cutting the copper strips parallel with the direction of rolling.

(*n*) *Resistance to Oil.* This test has been standardized as follows:[4] Specimens shall be prepared as described in (*m*) "Resistance to Heat." The effect of oil on the varnish shall be determined by immersing the specimens in transformer oil at a temperature of 105 to 110° C. (221 to 230° F.) for 48 hours and noting the effect on the varnish as indicated, for example, by wiping with a piece of dry white cloth.

NOTE. Incipient disintegration of the surface of the varnish may sometimes be detected by examining the oil for turbidity. If a specimen of the used oil filtered through filter paper can be distinguished from an unfiltered sample of the used oil, when the two samples in identical containers are held in front of a diffused light, the oil is turbid.

(*o*) *Resistance to Acids and Alkalies.* The dried film is subjected to the action of sulfuric acid, sp. gr. 1.25 (about 33 per cent), nitric acid sp. gr. 1.12 (about 20 per cent), hydrochloric acid sp. gr. 1.09 (about 18 per cent), and sodium-hydroxide sp. gr. 1.15 for a period of six hours, and the action noted.

(*p*) *Dielectric Strength.* A method for testing insulating varnishes has been standardized, the details of which will be found in the original publication.[4] Other investigations have been undertaken [12] to ascertain the relationship between the dielectric strength and the rust-inhibiting properties of bituminous-solvent compositions.

(B) ESTIMATION, RECOVERY AND EXAMINATION OF THE SOLVENT

Test 85. Estimation and Recovery of Solvent. Two methods have been adapted for this purpose, as follows:

(*a*) *Evaporation Method.* The method devised by A. L. Brown is rapid and gives accurate results, but does not recover the solvent for further examination.[13] Deliver 3–4 ml. of the well-mixed material (cements as well as paints of a heavy body should first be thinned to fluid consistency with a weighed quantity of pure benzol) from a 10-ml. pipette into a weighed glass flask of 50-ml. capacity, as rapidly as possible. Stopper the flask immediately, weigh, and dilute to the mark with pure benzol. Deliver exactly 10 ml. of the well-mixed material from the pipette upon a weighed ground-glass plate, 10 by 15 cm. and 1.5–3.0 mm. thick, supported in a level position. The diluted material should be flowed gradually on the plate, the object being to cover it entirely, without causing the solution to creep over the edges. It is recommended that 7 ml. be delivered first, and the remainder, a few drops at a time during the ensuing two minutes. The evaporation of the benzol will carry most of the solvent with it, and the film is so thin that the solvent will evaporate in one and one-half to two and one-half hours, the plate being weighed every half hour to follow the course of evaporation. Should the material contain a drying oil, the plate must be placed in an atmosphere of illuminating gas after the first half hour, replacing it after each weighing. The solvent has entirely evaporated when a constant weight is obtained. From this calculate the percentage of solvent by weight. An idea of the drying qualities of the film may be gained by placing the glass in a free circulation of air after the solvent is eliminated, and weighing it every hour as the film oxidizes, until it no longer *increases* in weight. If the coating has a tendency to dry unevenly, a weighed quantity of 50-mesh sea sand, previously dried and ignited, may be sifted over the paint in a very thin layer, but so the paint will be visible between the grains of sand. This will insure a uniform evaporation of the solvent.

An alternate procedure has been standardized as follows: The apparatus required consists of a chemical balance, a glass ther-

mometer having a range of 0 to 150° or 200° C. and accurate within 1° C., a constant-temperature oven of uniform temperature, a stoppered bottle and flat bottom metal drying dishes. Each metal drying dish shall have an inside diameter of approximately 2¾ in. (7 cm.) and a depth of approximately 5/16 in. (8 mm.).

NOTE. A standard single-friction tin can cover having a diameter of 2 13/16 in. is suitable for use as a drying dish.

The specimen shall consist of approximately 1.5 g. (1.35 g. to 1.65 g.) of the sample of varnish taken from the shipment. A portion of the sample of varnish shall be placed in a stoppered bottle or weighing pipette and weighed. Approximately 1.5 g. (1.35 g. to 1.65 g.) shall be transferred from the weighed stoppered bottle to a weighed drying dish which has previously been heated for 30 min. at 135° C. and cooled in a desiccator. The stoppered bottle with the remaining contents shall be weighed again. The exact weight of the specimen transferred to the drying dish shall be determined by difference. A total of three specimens shall be prepared from the contents of the stoppered bottle.

The specimens shall be placed in the constant temperature oven within 30 min. after preparation. The specimens shall be heated for a period of 3 hr. at the temperature designated, as follows:

Impregnating varnishes of the phenolic-resin type.....132 to 138° C.
Other types of insulating varnishes.....105 to 110° C.

NOTE. In a gravity type of oven which depends upon the natural circulation of air for uniformity of temperature, only one shelf must be used for supporting the specimens, and the bulb of the thermometer for indicating the temperature shall be in close proximity to the specimens.

At the termination of the 3-hr. heating period, the specimens shall be removed to a desiccator for cooling to room temperature. Each specimen shall be weighed immediately upon removal from the desiccator. The ratio of the weight of the dried specimen to the weight of the specimen in the original state expressed as a percentage is the percentage of non-volatile matter by weight.

The report shall include the following:

(*a*) The type of varnish;
(*b*) The oven temperature, and
(*c*) The percentage of non-volatile matter.

(*b*) *Steam Distillation Method.* This has been standardized as follows:[14] The bituminous mixture is distilled in a current of steam, the solvent is condensed and separated from the water. The steam-generator shall be made of either metal or glass, with a capacity of from 2 to 4 liters, suitable for continued use in the production of steam. If of glass, it shall be fitted with two outlets with suitable connections for rubber tubing. In the case of a metal generator, a large opening for filling and a water gage shall be additional parts of the apparatus. The generator shall be supplied with suitable pinch cocks or valves so that steam may be blown off to the atmosphere until the test is ready. The bath shall be of metal of sufficient capacity to permit immersion of the distilling flask to a depth of not less than 10 cm. Heat for the steam generator shall be supplied by a suitable gas generator or electric hot plate. The bath may be heated by any convenient means.

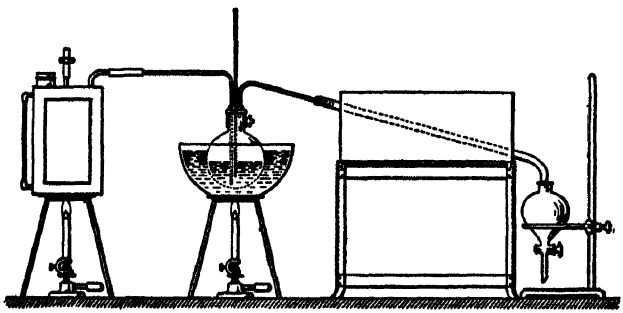
The distilling flask shall be a short ring-neck, round-bottom flask of 1000-ml. capacity. It shall be fitted with a three-hole rubber stopper; with a steam distilling tube which will reach to within ½ in. (12.7 mm.) of the bottom of the flask and project from the top at a convenient distance for connection to the generator; a vapor outlet tube which extends from beneath the rubber stopper to a point sufficiently above the distilling flask that will permit convenient connection to the condenser; and with a thermometer. The steam tubing should be not less than 2 nor more than 4 mm. in internal diameter and the vapor outlet tube should be not less than 5 mm. in internal diameter.

The condenser shall consist of a 9/16-in. (14.29-mm.) outside diameter No. 20 Stubbs' Gage seamless brass tube, 22 in. (55.88 cm.) long. It shall be set at an angle of 75 deg. from the perpendicular and be surrounded with a cooling bath 15 in. (38.1 cm.) long, approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube shall be cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to 1¼ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate.

A separatory funnel having a capacity of not less than 500 ml. should be provided. Accessories consist of suitable ring stands for

supporting the steam generator, distilling flask, bath for distilling flask, separatory funnel, and a thermometer.

The apparatus shall be assembled as shown in Fig. 317. The steam-generator shall be filled with water and heat applied. The bath shall be filled with a high-flash-point oil and raised to approximately 140° C. (284° F.). Five hundred milliliters of the sample shall be weighed into the round-bottom flask. The connection shall be made from the steam-generator to the steam delivery tube, the end of which shall be within ½ in. of the bottom of the distilling



Courtesy A.S.T.M.

FIG. 317.—Assembly of Distillation Apparatus.

flask. The outlet from the distilling flask shall be connected to the condenser and the separatory funnel placed in position at the outlet of the condenser to receive the distillate. The end of the bulb of the thermometer in the steam-distilling flask shall be placed within ½ in. (12.7 mm.) of the bottom of the distilling flask.

When the temperature of the sample in the distilling flask reaches 130° C. the outlet of the steam generator shall be closed, thus forcing the steam to pass through the sample. The flow of steam shall be adjusted so that the distillate is collected at the rate of approximately 6 to 10 ml. per minute. The distillation shall be stopped when 100 ml. of the distillate contains not more than 0.5 ml. of solvent, as determined by measuring the amount of oil in 100 ml. of distillate. When the distillation is finished, the water shall be separated from the distillate and the distillate measured and retained for further tests, if required by the specifications.

In some cases, the distillate does not separate readily from the water, and this separation can be facilitated by the addition of

sodium chloride, which will result in a sufficient difference in gravity to produce a clear separation of the two layers.

The results shall be reported in per cent by weight or volume as required by the specifications, based on the weight of the sample taken.

NOTE. In the distillation of varnishes the end-point differs with the various types and compositions. In all cases, however, the rate of distillation slows down as the end-point is approached; and, when the end-point is reached, acrolein (formed in the decomposition of the varnish base) is evolved and may be detected by its characteristic odor.

In addition to the evolution of acrolein, the following phenomena are suitable indicators of end-point:

(1) When the end-point of the solvent is considerably lower than the initial boiling-point of the base, the end-point of the distillation is indicated by the temperature reaching a maximum and then starting to fall consistently.

(2) When the end-point of the solvent and the initial boiling-point of the base are too close, or overlap, the above temperature drop does not occur. The end-point of the distillation is then indicated by a sudden foaming up of the residual base in the distillation flask; or, if the distillation has been accompanied by foaming, the end-point is indicated by a breaking of surface tension with a consequent cessation of foaming.

The specific gravity of the volatile matter (solvent) recovered by distillation shall be determined in accordance with the procedure described below. The difference between 100 per cent and the percentage of non-volatile matter determined above shall be calculated and recorded as the percentage of volatile matter by weight. The percentage of volatile matter by volume shall be calculated from the formula:

$$x = \frac{AC}{B}$$

where

x = the percentage of volatile matter by volume;
A = the specific gravity of the original varnish, as determined by Test 84 (*a*).
B = the specific gravity of the volatile matter (solvent) recovered by distillation, and
C = the percentage of volatile matter by weight.

With proper care and attention to detail in making this test, differences occurring between different laboratories should not exceed 2 per cent for varnishes containing solvents which have end-points not over 235° C. (455° F.), and should not exceed 4 per cent when the end-point of the solvent is as high as 307.2° C. (585° F.).

To correct the loss of distillate for water-soluble constituents, weigh out another 100 g. into a 250-ml. flask and distil without steam over an electric stove. Continue the distillation until the residue in the flask reaches a temperature of 200° C. This gives somewhat lower results than the first method, but the distillate should be tested for water-soluble substances to correct the results obtained by the previous method. Turpentine dissolves to the extent of 0.3 g. for each 100 ml. of water condensed.[15]

If the residue is to be used for further examination, any water introduced into it by the steam distillation may be removed by distilling it twice with ten times weight of a mixture of 3 parts benzol and 1 part alcohol.[16]

Test 86. Examination of the Solvent. The solvent recovered from Test 85 should be tested for specific gravity, refractive index, flash-point,[17] distillation range,[18] percentage unacted upon by concentrated sulfuric acid, etc., to assist in its identification.[19]

(C) ESTIMATION, RECOVERY AND EXAMINATION OF PIGMENT AND FILLER

Test 87. Estimation and Recovery of Pigment and Filler. Dilute 100 g. of the well-mixed material with 500 ml. of benzol in an 800-ml. stoppered flask. Either centrifuge or let stand in a warm place until the pigment or filler has settled, then carefully decant the supernatant liquid into a clean flask of large capacity. The pigment or filler is shaken up with 250 ml. more benzol, allowed to stand in a warm place until it settles, and the supernatant liquid decanted into the second flask. Repeat the treatment with benzol until the vehicle has been completely extracted from the pigment. The prevention of skin formation during this process may be attained by the addition of anti-oxidants such as phenol or hydrochinone (dissolved in ether),[20] which are subsequently expelled on heating the pigment and base. The combined extracts are allowed to stand quietly to recover any pigment that may have been carried over with the benzol, and then carefully decanted through a weighed Gooch crucible provided with an asbestos mat. The residues in the flask and on the Gooch crucible are washed with benzol as before,

and combined with the balance of pigment or filler, which is then dried at 110° C. and weighed. The combined extracts are retained for further examination.

Test 88. Examination of the Pigment or Filler. The pigments or filler recovered from the Test 87 are subjected to a qualitative or quantitative analysis for purposes of identification,[21] likewise to granularmetric tests.[22]

(D) ESTIMATION, RECOVERY AND EXAMINATION OF THE BASE

Test 89. Estimation and Recovery of the Base. If no pigments or fillers are present, the base is recovered as described in Test 85, and its percentage by weight ascertained directly. If pigments or fillers are present, the combined extracts from Test 87 are distilled to a small bulk, transferred to a tared dish, and evaporated in an oven at 110° C. *exactly* to the calculated weight of the base, by subtracting the weights of solvent and pigment or filler from the original weight of material taken for examination. When oxidizable substances are present, the final evaporation should take place in an atmosphere of illuminating gas.

Test 90. Examination of the Base. The base will contain the bituminous constituents (with the exception of any "free carbon" associated with coal-tar pitch, or the like, which will be separated with the pigments and fillers), likewise animal and vegetable oils or fats, resins, and metallic bases and driers. It may be separated into its component parts in accordance with the procedure tabulated on the next page.

The following is an outline of a method devised by the author for examining the dry films of lacquers, cements, varnishes, enamels, or japans which have been applied to surfaces of metal, wood, masonry, or prepared roofing. It often happens that none of the original material is available, and it becomes necessary to examine the paint after it has been applied to the object intended, and allowed to harden or oxidize in the air, either at normal or elevated temperatures. The following method has been found to yield fairly accurate results.

Carefully scrape 50.00 g. of the lacquer or varnish film from the surface to which it has been applied, and avoid including any of

Method of Analyzing the Separated Base

Dissolve 50 g. in 150 ml. benzol. Add 10 ml. dil. nitric acid (1 : 1) and boil under a reflux condenser for one-half hour to decompose any metallic soaps (i.e., driers, etc.). Add 150 ml. water, boil under reflux condenser, transfer to a separatory funnel, draw off the aqueous layer, boil with another 100 ml. water, and repeat if necessary until all the metals are removed.

Benzol Solution:
Distil to 100 ml., add 300 ml. of the saponifying liquid (Test 37*e*), boil under reflux condenser for one hour, and separate the unsaponifiable and saponifiable constituents as described in Test 37*e*.

Unsaponifiable Matter:
Examine a small portion by Test 41. If higher alcohols are present, separate the balance by Test 37*f* into:

Hydrocarbons:
Contain the bituminous substances (i.e., asphalt, coal-tar pitch, unsap. matter derived from fatty-acid pitch, etc.)
Examine by the methods included in Chap. XXXII.

Higher Alcohols, Etc.:
Contain cholesterol, etc., derived from wool grease, also the unsaponifiable constituents originally present in resins (4 to 8%).

Saponifiable Matter:
Separate the fatty and resin acids as described in Test 37*g*.

Fatty Acids:
Include acids derived from vegetable and animal oils or fats, also from fatty-acid pitch.
(Note "A")

Resin Acids:
Include acids derived from rosin and the fossil resins.
(Note "B")

Aqueous Layer:
Determine percentage glycerol by Test 37*h*. Multiply this by 10 to estimate per cent of vegetable or animal oils or fats (triglycerides) present in the original substance.
(Note "C")

Aqueous Extract:
Contains the metallic bases as nitrates. Examine qualitatively and then quantitatively for lead, manganese, cobalt,[23] zinc, calcium, and magnesium.
(N. B.—The last three used for hardening rosin. The metallic dryers should *not* be found by ignition, since the lead will be reduced to metal by the organic matter, and volatilize.)

Note "A"

The following means are used to distinguish between the fatty acids derived from oxidized vegetable or animal oils and fatty-acid pitch respectively:

	Fatty Acids Derived From Vegetable or Animal Oils	*Fatty Acids Derived From Fatty-acid Pitch*
Lactone Value (Test 37*b*)	Less than 25	Greater than 25
K. and S. Fusing-point (Test 15*a*)	Less than 80° F.	Greater than 80° F.
Hardness at 77° F. (Test 9*c*)	Less than 5.0	Greater than 5 0
Color in Mass (Test 1)	Translucent yellow to brown	Opaque brown to black

Note "B"

Test qualitatively for rosin by the Liebermann-Storch reaction (Test 41). Fossil resins may be distinguished from rosin by determining the saponification, acid and ester values of the mixed resin acids. The following figures have been reported on the resin acids separated as described:[24]

	Saponification Value	Acid Value	Ester Value
Straight rosin varnish	182–185	160–162	22–24
Rosin ½; Kauri ½ varnish	122–135	44–62	72–78
Rosin ½; Kauri ½ varnish	143.5	88	55.5
Straight Kauri varnish	130	45	85
Untreated rosin	165–180	155–170	0–13
Untreated Kauri gum	124	41	83

Other resins may be examined in a like manner, but unfortunately, figures are not at present available.

Note "C"

If this corresponds with the total saponifiable matter present, then fatty-acid pitch and resins are absent.[25]

the underlying surface.* From this point on the method is outlined in the following table:

METHOD OF ANALYZING DRIED FILMS

Boil 50 g. scrapings with 350 ml. of the saponifying liquid (Test 37*e*) under a reflux condenser for one hour. Add 300 ml. benzol-alcohol (1 : 1), boil, let settle and decant the supernatant liquid into a large flask. Repeat the treatment with benzol-alcohol (1 : 1) until most of the soluble constituents have been extracted, then combine the extracts and let stand quietly to recover any further settlings, which after decantation and washing are added to the main portion of the residue.

<table>
<tr><td>Benzol-Alcohol (1 : 1)
Extracts:</td><td colspan="3">Residue:
Dry in an oven at 100° C., pulverize finely, transfer to a paper thimble and extract in a Soxhlet with benzol-alcohol (2 : 1) for 12 hours.</td></tr>
<tr><td></td><td colspan="2">Benzol-Alcohol (2 : 1) Extract:
Combine the benzol-alcohol (2 : 1) and (1 : 1) extracts, evaporate to a small bulk, and separate the unsaponifiable and saponifiable constituents as described in Test 37e.</td><td>Residue:
Dry, ignite and weigh. This includes pigments and fillers, free carbon from tars or pitches, also any metallic dryers present (Note "A").</td></tr>
<tr><td>Unsaponifiable Matter:
Examine as described.</td><td>Saponifiable Matter:
Examine as described.</td><td>Aqueous Layer:
Examine as described.</td><td></td></tr>
</table>

Note "A"

This should be examined microscopically for fillers, and subjected to a qualitative or quantitative chemical analysis. Note that any chrome green, chrome yellow, Prussian blue, etc., are transposed by the alkali, and these, also lampblack or carbon blacks are decomposed on ignition, for which due allowance must be made.

* The blade of a safety razor held at right-angles to the surface scraped, and drawn across it slowly but firmly has been found convenient for this purpose.

CHAPTER XXXVI

EXAMINATION OF BITUMINOUS DISPERSIONS

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT

Test 91. Method of Identification. The types of bituminous dispersions include the following:[1]

I. Quick-setting dispersions (i.e., those which set rapidly upon being mixed with mineral aggregate).

II. Slow-setting dispersions (i.e., those which set slowly upon being mixed with mineral aggregate). These in turn comprise:

(*a*) Semi-stable dispersions (i.e., those in which the breaking is retarded through the action of added "stabilizers").

(*b*) Stable dispersions, in which the setting results:

1. Mainly through evaporation of the water and to a slight extent by breaking.
2. Entirely through evaporation of the water content.

The foregoing types may be identified as follows:[2] If necessary, dilute with distilled water until the content of bituminous matter is within 50 to 52 per cent by weight. Add two times the volume of 95 per cent ethyl alcohol.

If the bituminous matter remains in suspension = Type II-*b*-2.
If the bituminous matter separates as a gummy mass = Types I, or II-*a*, or II-*b*-1.

Dilute 10 ml. of the dispersion with 25 ml. distilled water and gradually add 20 ml. N/50 $CaHCO_3$ solution. Shake vigorously for 1 minute and heat to incipient boiling, thereby causing the bituminous constituents to settle out and adhere to the sides of the container. Cool and pour the liquid portion into a clean container, washing out the first container with two 5 ml. portions of distilled water. To the combined extracts, add slowly from a burette 0.1 per cent aqueous solution of Neptune Blue R. G. (manufactured

by I. G. Farbenindustrie A.–G.) until the brown color of the liquid turns to an olive green or greenish tone.

If less than 10 ml. of the dye solution is required = Type I.
If from 10 to 30 ml. of the dye solution is required = Type II-*a*.
If more than 30 ml. of the dye solution is required = Type II-*b*-1.

Test 92. Homogeneity. The following tests have been proposed:

Test 92a. Appearance Under Microscope. After diluting with a weak solution of NaOH (to prevent coagulation), transfer to a glass slide under a cover-glass, and measure the size of the particles under a magnification of 500 to 600 diameters. The particles should not exceed 5 μ in diameter. Typical fields of a well-prepared dispersion are illustrated in Figs. 318(a) and 318(b), the first under a magnification of 600 diameters, and the second a view of the same dispersion under a magnification of 1200 diameters.

Test 92b. Sieve Test. Coarse particles and lumps may be ascertained in the following manner:[8]

The following apparatus and reagents will be required:

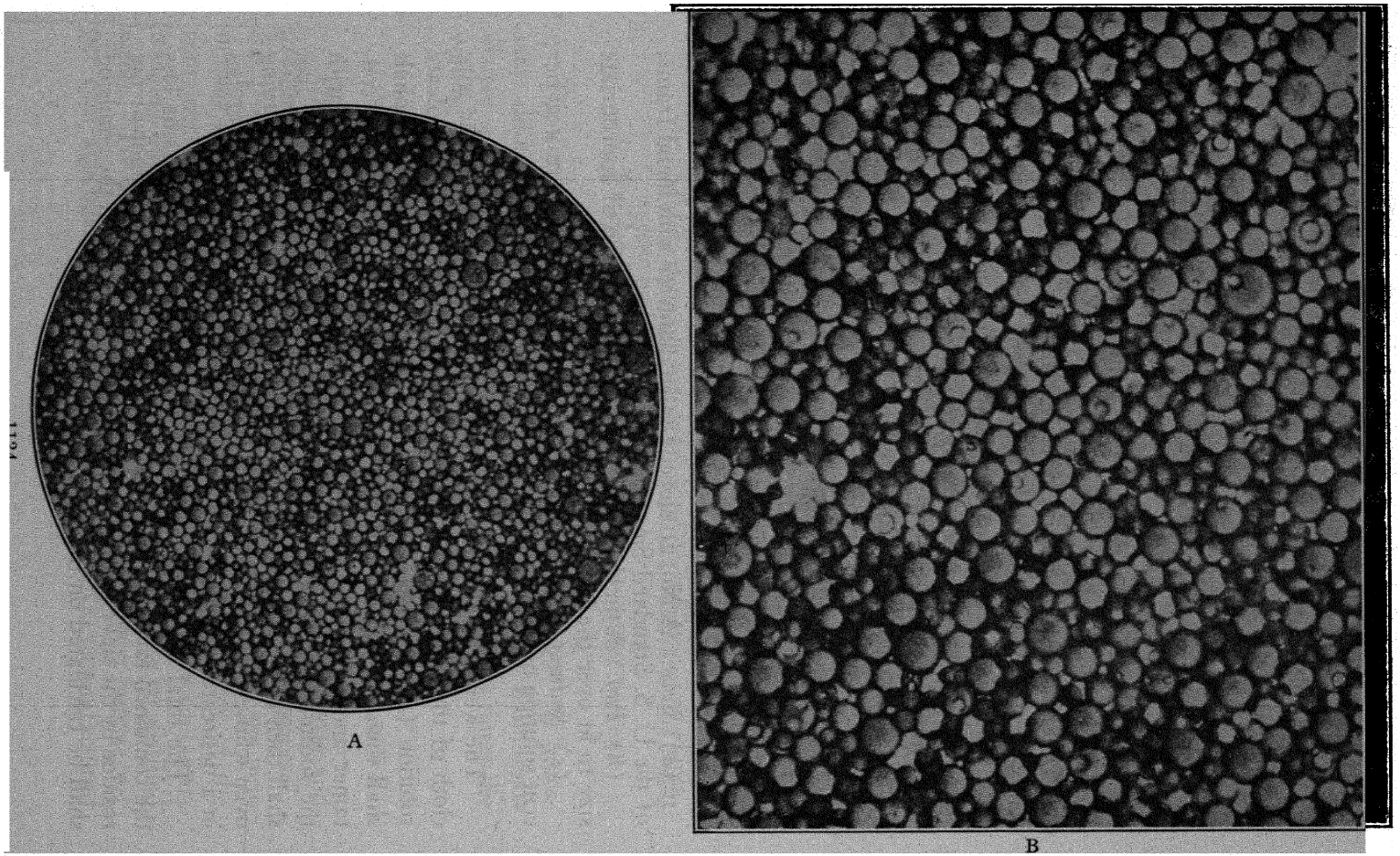
(*a*) *Sieve.* A No. 20 sieve, 3-in. in diameter with metal frame, of the U. S. Standard Sieve Series.

(*b*) *Pan.* A tin box cover or shallow metal pan of appropriate size to fit over the bottom of the standard sieve.

(*c*) *Sodium Oleate Solution* (*2 per cent*). Prepare a 2 per cent solution of pure sodium oleate in distilled water.

The weight of the sieve and pan shall be recorded and the wire cloth of the No. 20 sieve shall then be wet with the sodium oleate solution (2 per cent). Exactly 1000 g. of the emulsified asphalt shall then be weighed and poured through the wire sieve, the container and the residue on the sieve being washed thoroughly with the sodium oleate solution until the washings run clear. The pan shall then be placed under the sieve and heated for 2 hr. in a drying oven whose interior temperature is 220° F. (105° C.), then cooled in a desiccator and weighed.

The total weight of the sieve, pan, and residue in grams less the combined tare weight of the sieve and pan, is the weight of the residue by the sieve test. The percentage of residue in the emulsion shall be calculated on the basis of this weight.



Figs. 318A and 318B.—Typical Bituminous Dispersions in Water.

Test 92c. Settlement Test. This is performed as follows:[4] The following apparatus will be required:

(*a*) *Cylinders.* Two glass cylinders of 500-ml. capacity with pressed or molded glass base and cork or glass stoppers. The outside diameter shall be 5.0 cm. ±0.5 cm. and the cylinders shall be graduated at each 5-ml. interval to the 500-ml. mark.

(*b*) *Glass Pipette.* A syphon, glass tube pipette, 60-ml. capacity, form optional.

A 500-ml. sample, representative of the emulsion, shall be placed in each of two glass cylinders. The cylinders shall be stoppered air-tight and stood aside unmolested, at laboratory air temperature for 5 days. After standing for this 5-day period, approximately the first 55 ml. of emulsion shall be removed by means of the pipette or syphon from the top of each sylinder without disturbing the balance of their contents. Exactly 50 g. of each of the two samples, after each has been thoroughly mixed separately, shall be weighed into separate 600-ml. low-form glass beakers and the asphaltic residue determined by evaporation at 325° F (163° C.) for 3 hr. in the apparatus described in Test 16.

After removal of the first sample, approximately the next 390 ml. shall be syphoned off from each of the cylinders. The residue remaining in the cylinders shall be mixed thoroughly and exactly 50 g. shall be weighed out from each of them and the amount of asphaltic residue (all sediment, if any, included) shall be determined by evaporation as described above for the two top samples.

The numerical difference between the average percentage of asphaltic residue from the two top samples and the average percentage found in the two bottom samples shall be recorded.

Test 92d. Stability on Ageing. The dispersion is allowed to stand in a tightly closed container and retested as in 92b and 92c at the end of 1, 3 and 6 months respectively, to ascertain whether it has a tendency to separate or "reverse" upon standing.

Test 93. Viscosity. The viscosity determination shall be made at 77° F. (25° C.) and shall be expressed in seconds, Saybolt Furol, being the time in seconds for the delivery of 60 ml. of emulsion. While the Saybolt Furol viscosimeter is not used for petroleum products and lubricants when the time of flow is less than 25 sec.,

this instrument is satisfactory for testing emulsified asphalt when the time of flow is not less than 20 sec.

The sample shall be stirred thoroughly without incorporating bubbles in it and then poured into a 4-oz. bottle. The bottle shall then be placed in the water bath at 77° F. (25° C.) for 30 min. and the sample then mixed in the bottle by inverting several times, slowly enough to prevent bubble formation. The sample shall then be poured into the viscosity tube through a 20-mesh strainer, allowing a small portion to flow through the outlet tube to waste. The cork shall then be placed in position, the tube filled and without again stirring the sample the viscosity shall be determined as described in Test 8b.

Test 94. Demulsibility. This test indicates the rapidity with which the dispersion "breaks" during use, and may be ascertained in accordance with the following procedures:

Test 94a. Calcium-chloride Test: [5]

The following apparatus and reagents will be required:

(*a*) *Sieves.* Three No. 14 sieves of the U. S. Standard Sieve Series, of iron wire cloth, unframed, approximately 5-in. square.

(*b*) *Beakers.* Three glass beakers of 600-ml. capacity each.

(*c*) *Glass Rods.* Three glass rods, rounded ends, approximately 5/16 in. in diameter.

(*d*) *Burette.* A 50-ml. glass burette graduated in 0.1 ml.

(*e*) *Calcium Chloride Solution (0.02 N).*

(*f*) *Calcium Chloride Solution (0.10 N).*

The percentage of residue shall be determined by distillation as described in Test 98.

The weight of each assembly of beaker, rod and sieve shall be recorded. Exactly 100 g. of the emulsified asphalt shall be weighed into each of three 600-ml. tared beakers. Over a period of approximately 2 min., 35 ml. of 0.02 *N* $CaCl_2$ solution (if quick-setting emulsion is being tested) or 50 ml. of 0.10 *N* $CaCl_2$ solution (if mixing type emulsion is being tested) shall be added to each beaker from a burette. While adding the solution of $CaCl_2$, the contents of the beaker shall be stirred continuously and vigorously, kneading lumps against the sides of the beaker to insure thorough mixing of the reagent with the emulsion. This operation shall be performed after bringing the weighed sample of emulsion and the reagent to

the standard temperature of 77° F. ± 1.0° F. (25° C. ± 0.5° C.).

One of the wire sieves shall be fitted over a beaker or other suitable vessel and the mixture of emulsion and reagent poured through the sieve. The beaker, containing the sample and glass rod, shall be rinsed with distilled water. All lumps shall be kneaded and broken up and the washing of the beaker, rod and sieve shall be continued until there is no longer any appreciable color imparted to the wash water. After washing as directed, the beaker, rod and sieve used in each individual test shall be placed in a drying oven and dried at 325° F. (163° C.) to constant weight.

The total weight thus obtained less the total tare weight of the beaker, rod and sieve shall be the weight of the residue by the demulsibility test. The ratio of the average weight in grams from three tests of each individual sample of emulsified asphalt, A, to the weight in grams of residue per 100 g. of emulsion, B, obtained in the test for residue by distillation described in Test 98 multiplied by 100, shall be recorded as the percentage of demulsibility of the sample tested:

$$\text{Percentage Demulsibility} = \frac{A}{B} \times 100$$

Test 94b. Ferrous-Sulfate Test. This test is claimed to be more severe than the Calcium-chloride Test, and is determined as follows: 50 g. of the dispersion are weighed into a 250 ml. beaker, diluted with 25 ml. distilled water and 10 ml. of a freshly prepared 10 per cent $FeSO_4 \cdot 7H_2O$ solution added during a period of approximately 2 minutes, the contents of the beaker being continuously stirred with a glass rod. The solution is diluted to 100 ml. and permitted to stand for 24 hours, when an additional 100 ml. of distilled water are added. The contents of the beaker are then drained through a 40-mesh sieve and the unbroken emulsion in the beaker and on the rod is rinsed through the sieve with water until there is no appreciable discoloration of the rinsing water. The beaker, rod and sieve are permitted to dry at room temperature and weighed every 24 hours until constant weight is attained. The weight of residue deposited on the beaker, rod and sieve is recorded as an index of the demulsibility.

Test 94c. Behavior with Aggregate ("Coating Test"). This test is performed in the following manner:[6]

NOTE. This test is applicable only for emulsions containing a base of asphalt of semi-solid consistency. It is not applicable to the so-called quick-setting type of emulsions.

The following apparatus and reagents will be required:

(*a*) *Screens.* A standard ¾-in. screen and ¼-in. screen conforming to the requirements of the Standard Specifications for Round-Hole Screens.

(*b*) *Spatula.* A steel spatula or its equivalent, blade approximately 8 in. in length.

(*c*) *Dish.* A round-bottom, iron dish or a kitchen saucepan, approximately 1-qt. capacity.

A supply of reference stone (hard limestone, trap rock, or other type) which has been washed with water and dried before using. The grading of this stone shall be such that it will all pass through a standard ¾-in. screen and not more than 5 per cent will pass through a ¼-in. screen.

NOTE. Each laboratory shall select its own reference stone supply, the source of which is not apt to change. This is to obviate rapid changes in the character of reference stone used in any one laboratory.

Exactly 465 g. of the washed and dried graded stone shall be weighed and placed in the metal pan. A 35-g. sample of the emulsion shall then be added to the stone in the pan and mixed vigorously with the spatula for 3 min.

Record whether or not there is appreciable separation of the asphaltic base from the water of the emulsion and whether or not the stone is uniformly and thoroughly coated with the emulsion.

The foregoing demulsibility tests will indicate whether the emulsions are of a slow-setting type or a quick-setting type.[7] Slow-setting emulsions, otherwise termed "mixing emulsions" must be capable of dilution with water, and when used for coating aggregates by machine or hand-mixing methods must permit a uniform distribution and of handling and manipulation of the mixture incident to construction. Quick-setting emulsions, otherwise termed "penetration emulsions" must coalesce or demulsify as soon as they come in contact with the aggregate when they are spread over the pavement already laid in place. Slow-setting emulsions contain considerable quantities of dispersing agents, and set largely by evaporation of the water present. Quick-setting emulsions contain either

very small quantities of the dispersing agent, or a small quantity of a previously formed quick-setting emulsion.[8]

Test 95. Miscibility with Water. This test is performed as follows:[8]

NOTE. This test is not applicable to the so-called quick-setting type of emulsions.

To about 50 ml. of the emulsion shall be gradually added about 150 ml. of distilled water, stirring the mixture while adding the water. The temperature is not important but should be between 21 and 25° C. (70 and 77° F.). The mixture shall be allowed to stand for 2 hr. and then examined for any appreciable coagulation of the asphalt content of the emulsion.

Test 96. Effects of Freezing. This test has been standardized as follows:[8]

Approximately 400 g. of the emulsion shall be placed in a clean metal container, such as a 1-pt. press-top tin. The emulsion in the closed container shall be exposed to a temperature of 0° F. (−17.7° C.) for twelve consecutive hours. At the expiration of the freezing period, the emulsion shall be permitted to thaw by exposure of the container to the temperature of the laboratory.

After the first operation of freezing and thawing, the procedure shall be repeated twice, so that the emulsion will have been subjected to three cycles of freezing and thawing.

After the third cycle, the emulsion may be homogeneous or may have separated into distinct layers which cannot be rendered homogeneous by stirring at laboratory temperature.

The result of this test shall be reported as either "Homogeneous" or "Broken."

Test 97. Resistance to Water after Setting.

(*a*) *Without Aggregate.* A slight excess of the emulsion (thinned with water to the consistency in which it is recommended to be used) is poured into a clean porcelain dish which by suitable manipulation is caused to flow over the entire inner surface. The excess is then allowed to drain out. The dish is then stood upright at room temperature and observed from time to time until the film has dried, which may generally be ascertained when it turns from a brown to a black color. The time of drying is recorded, whereupon the dish is maintained at room temperature for twenty-four hours.

At the termination of this period it is filled with distilled water and examined at the end of one, two, four and eight hours respectively, to determine whether or not the film is softened or dissolved by the water.

(*b*) *When Mixed with Aggregate.* Clean, rounded river gravel of uniform quality from a source permitting of duplication for future work should be selected. Two 1000 g. samples passing a ½-in. and retained on a ¼-in. screen are placed in wire baskets and dried to a constant weight in an oven at 125° F. The baskets are immersed for two minutes in the emulsion to be tested. The emulsion and aggregate are maintained at 77° F. during the test and the room at 77° F. at a relative humidity of about 50. After removal from the emulsion, one basket is allowed to drain exactly 30 minutes and is then washed with tap water for 15 minutes, by allowing a stream of water to flow through a ¼-in. hose at the rate of 1 gallon per minute until the water comes through clear. The basket and contents are dried to a constant weight in an oven at 120° F. The increased weight due to the deposited asphalt is recorded. The other basket is dried to a constant weight in the same manner, without spraying with water. The asphalt remaining on the aggregate in the washed basket is calculated as a percentage of the asphalt deposited on the aggregate in the unwashed basket.

(B) SEPARATION OF THE DISPERSION INTO ITS COMPONENT PARTS

The following products are likely to be present, viz.: water, ammonia, various chemicals, bituminous matter, animal and vegetable oils or fats, other forms of non-bituminous organic matter and mineral matter.

Test 98. Distillation Residue. The following method has been standardized:[8]

The following apparatus will be required:

(*a*) *Iron Still.* The iron still shall be approximately 6 by 3½ in. in inside diameter with adjustable ring burner with holes on the inner periphery to fit around the outside of the still.

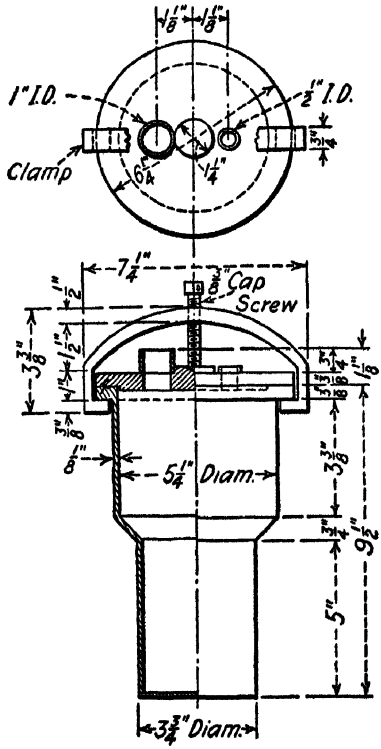
NOTE. A modification of this still is shown in Fig. 319. It consists of the regular still with an expansion chamber superimposed thereon. Two additional ring burners are required, one approximately 6 in. in inside diameter with the holes bored on the inside periphery, and one approximately 2 in. in diameter with the holes bored on top.

(*b*) *Connecting Apparatus.* A connecting tube, tin shield, condenser trough, condenser tube and graduated cylinder, as shown in Fig. 320.

(*c*) *Thermometer.* A thermometer graduated from 30 to 580° F. (0 to 300° C.).

NOTE. The details of the assembly of apparatus for the distillation test are illustrated in Fig. 320.

Exactly 200 g. of a well-mixed and representative sample of the emulsion shall be placed in the previously weighed iron still including lid, clamp, thermometer and gasket, if gasket is used). A gasket of oiled paper may be used between the still and its cover or the joint ground to a tight fit. The cover shall be clamped securely on the still. The thermometer shall be inserted through the small hole in the cover, using a cork stopper, so that the end of the bulb is ¼ in. from the bottom of the still. The ring burner shall be placed around the still and the heat applied by this means to the top of the still. Just enough heat from a Bunsen burner shall also be applied to the connecting tube to prevent condensation of water in this tube.

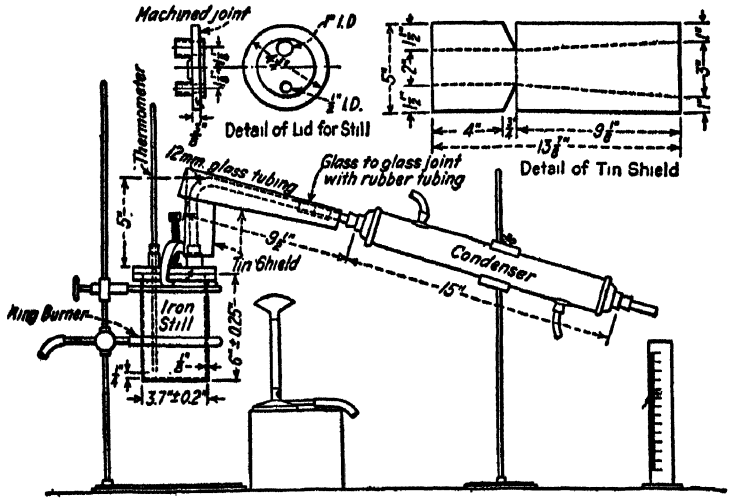


Courtesy A.S.T.M.

FIG. 319.—Iron Still for Use with Badly Foaming Emulsions.

After practically all the condensate has been removed from the still and the temperature of the residue has reached 250° F. (121° C.), the position of the heat from the ring burner shall be lowered to midway of the still and held there until the thermometer reaches 349° F. (176° C.). The burner shall then be rapidly lowered to within ¼ in. of the bottom of the still and the temperature increased to and maintained at 500° F. (260° C.) for 15 min. This latter period of heating is necessary to insure a smooth homogeneous residue in the still. At the expiration of the heating period at

the maximum temperature, the still and accessories shall again be weighed and the percentage residue calculated and reported. The cover shall then be removed from the still and suitable portions of the residue shall be poured immediately into suitable molds and containers for making the required tests. The residue in the molds and containers shall be permitted to cool, uncovered, to laboratory room temperature and thereafter tested for specific gravity (Test 7), penetration (Test 9), ductility (Test 10), fusing-point (Test 15), solubility in carbon disulfide and ash (Test 21).



Courtesy A.S.T.M.

FIG. 320.—Apparatus Assembly for Distillation Test of Emulsified Asphalts.

It should be noted that the distillation residue will carry any non-volatile dispersing agents present in the emulsion.

NOTE. When it appears impossible to distil an emulsified asphalt in the still described above due to excessive foaming of the emulsion, then the modified still shown in Fig. 319 should be substituted for the still shown in Fig. 320 and the following procedure followed: Place the 6-in. burner around the larger diameter of the still near its top. This serves as a support. Place the 4-in. burner immediately beneath the flare and the 2-in. burner not less than 2 in. below the bottom. Distillation is started with only the 2-in. burner lighted. Practically all of the distillate should be over in about 45 min. When the distillation apparently stops, light the two larger ring burners and adjust to a low flame. Distillation resumes and when it stops again increase the heat by adjusting the flame of the 2-in. burner. When the temperature can be read upon the thermometer, increase the rate of heating by raising the flame on both the 2- and 4-in. burners and bring the temperature to 500° F. (260° C.). If any evidence is noted of the emulsion beginning to foam over in the delivery tube, remove the 2-in. burner quickly and raise a pan of water so as to immerse the still bottom to a depth of about 2 in. for a moment which will check the foaming. Upon resumption of heating watch delivery tube carefully and repeat treatment if necessary.

When the residue has reached and remained at 500° F. (260° C.) for 15 min., proceed as described above for the regular still. While the distillation should be completed in not less than 1 hr. nor more than 1¼ hr. from the first application of heat to the still, the maximum stated is not mandatory as a longer time may be required in some cases to avoid foaming of the emulsion into the condenser. If the residue in the still prior to pouring the ductility and penetration specimens appears granular or heterogeneous in any way, stir with a spatula until the material runs from the spatula in strings instead of drops, and then pour. Should the residue then appear granular and heterogeneous, the test shall be rejected and the distillation repeated as previously described with the exception that the still temperature at 500° F. (260° C.) shall be maintained for a longer time than 15 min. until the desired condition of the residue is obtained.

Test 99. Water and Volatile Oils. The following method has been standardized:[9]

This method of test determines water existing in a sample of bituminous emulsion by distilling the sample with a volatile solvent.

The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method.

(*a*) *Metal Still.* The metal still, Fig. 283(a), shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

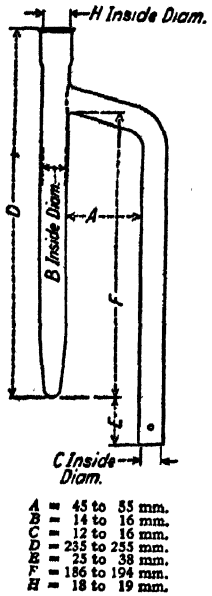
(*b*) *Glass Still.* The glass flask, Fig. 283(b), shall be of the short neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

(*c*) *Heat Source.* The burner used with the metal still shall be a ring gas burner 100 mm. (4 in.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

(*d*) *Condenser.* The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. (15¾ in.) in length with an inner tube 9.5 to 12.7 mm. (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 deg. from the vertical axis of the condenser.

(*e*) *Trap.* The trap shall be made of well-annealed glass con-

structed in accordance with Fig. 321 and shall be graduated from 0 to 25 ml. in 0.1-ml. divisions. The tolerance of the graduations between 0 and 2 ml. shall be ± 0.5 ml. and between 2 and 25 ml. shall be ± 0.1 ml. The outside diameters should be preferably 2.5 to 3.5 mm. (3/32 to 1/8 in.) greater than the inside diameters specified.



A = 45 to 55 mm.
B = 14 to 16 mm.
C = 12 to 16 mm.
D = 235 to 255 mm.
E = 25 to 38 mm.
F = 186 to 194 mm.
H = 18 to 19 mm.

FIG. 321.—Apparatus for Determining Water.

The solvent used when testing bituminous emulsions shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements (A.S.T.M. Designation: D 86):

98 per cent shall distil between 248° F. (120° C.) and 482° F. (250° C.).

The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE. The difficulties in obtaining proper representative samples for this determination are unusually great, so that the importance of sampling cannot be too strongly emphasized.

When the material to be tested contains less than 25 per cent of water, exactly a 100-g. sample shall be placed in the still or flask. When the material contains more than 25 per cent of water, the sample shall be 50 g. The sample to be tested shall be thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material.

The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown in Fig. 283 (*a*) and (*b*). The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. (0.04 in.) below the surface of the liquid in the trap after distillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per sec. The ring burner used with the metal still shall be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds. The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than 1 hr. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the weight of sample used, shall be the percentage of water and shall be reported as "..... per cent water by weight, A.S.T.M. method."

The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

The sum of the percentages of Distillation Residue and Water, deducted from 100 represents the percentage of Volatile Oils.

A rapid method for determining the amount of water present in the dispersion consists in weighing 50 g. of the well-mixed material into a standard 3-oz. tin box, which is then heated in an oven at 163° C. (325° F.) for five hours, and the loss in weight determined. Another rapid method consists in precipitating the bituminous constituents from the emulsion with hydrochloric acid, neutralizing with ammonia, and then pressing out the water mechanically, finally weighing the residue. Still another rapid method[10] for separating the water and soluble dispersing agents from the bituminous constituents and insoluble dispersing agents, consists in pouring 15 g. of the well-mixed emulsion onto a porous clay plate, which is then exposed to air at room temperature for 48 hours. The film is then scraped off, weighed, and tested for fusing-point, etc. It is claimed that in this procedure, the composition of the material is not altered in any respect.

Test 100. Dispersing Agents. These may be ascertained as follows:[11] Soaps are determined by adding 25 ml. of 96 per cent ethyl alcohol gradually to a 10-g. sample of the emulsion, and washing the separated residue with 10 ml. alcohol. Small quantities of

the bituminous constituents dissolved in the combined alcoholic extracts are removed by adding 20 ml. of water and then shaking with benzol in a separatory funnel. The separated bituminous constituents and benzol extract are then evaporated together and weighed. The bituminous residue is then tested for tar products. The alcoholic extracts are evaporated dry and the soaps are extracted with hot water and transposed with HCl. They may be tested for resin-, naphthenic- and fatty-acids as follows: resin acids are detected by the usual color reaction; naphthenic acids react with formaldehyde and sulfuric acid, forming an ether-insoluble formolite; whereas fatty-acids yield an ether-soluble formolite. The copper salts of naphthenic acids dissolve in benzol, forming a green-colored solution. Ammonia soaps may be detected by adding NaOH and heating. If present, ammonia may be determined quantitatively by distilling a weighed quantity of the emulsion into a standard solution of sulfuric acid and titrating with alkali. Sulfite liquor is extracted from the original emulsion with an equal volume of 70 per cent ethyl alcohol, whereupon the residue obtained on evaporation yields lignosulfonic acid with dilute HCl (which evolves SO_2 on heating), contains calcium, reduces Fehling's solution, and gives the carbohydrate reaction with α-naphthol and sulfuric acid. Inorganic dispersing agents may generally be determined by boiling the emulsion with dilute hydrochloric acid and extracting the bituminous substances with benzol. The inorganic chemicals will remain in the aqueous layer and may be determined by a qualitative or quantitative analysis. In other cases inorganic agents, such as clay, may be ascertained by incinerating a weighed quantity of the emulsion, and examining the ash. Special methods must be used for determining albumenoids, proteins, gums, alginates, alkaline bases, tannins, polysaccharides, sulfonated oils, etc.

CHAPTER XXXVII

WEATHERING TESTS

Effects of Weathering. All substances undergo a change on being exposed to air, moisture and sunlight. Metals undergo corrosion, rocks disintegrate, wood decays and animal or vegetable fibers decompose by hydrolysis. Bituminous substances are not immune from such action. On exposure to the weather (i.e., air, sunlight and moisture) they will change physically and chemically.

In the early days of photography, solutions of asphalt in ethereal oils such as turpentine, oil of lavender, etc., were used for preparing the sensitized photographic film. On exposure to light under the lens of a camera, certain changes took place in the asphaltic coating, as evidenced by the fact that upon subjecting it to the action of certain solvents, those portions which had been in contact with light became insoluble, whereas those protected from its action readily dissolved in the solvent, bringing the photographic image into relief. It took rather a long exposure to produce satisfactory images, since asphalt is only moderately sensitive to light, in comparison with some of the high-speed photographic plates in use at the present time. Nevertheless very artistic results have been produced by this crude method, which was originally discovered in 1816 by Joseph N. Niepce, who used a solution of asphalt in "Dippel's oil" obtained as a distillate in the destructive distillation of bones. In other experiments, he used oil of lavender and petroleum distillates to remove the asphalt unaffected by the light, followed by a final washing with warm water.[1]

It was soon observed that certain forms of asphalt were more photo-sensitive than others, and Syrian asphalt in particular became very popular on account of its purity, solubility, hardness and sensitiveness to the light's rays.[2] It was subsequently found that the addition of sulfur chloride increased the sensitiveness of native asphalts,[3] but petroleum asphalts were apparently rendered inert in its presence. Further investigations revealed the fact that petro-

leum asphalts free from paraffin are relatively the most sensitive towards light,[4] likewise mixtures of asphalt and rubber or gutta percha.[5]

Maximilian Toch noted that bituminous materials on exposure to sunlight decomposed with the liberation of "free carbon."[6] His experiments indicated that this action was inhibited by incorporating an opaque pigment. He pointed out further, that animal and vegetable oils (triglycerides) are not affected in this manner, and when blended with bituminous materials, apparently retard the action. Archaeological excavations indicate that asphalt coatings undergo no appreciable deterioration during thousands of years in the dark, but on exposure to light, rapid surface changes occur.[7]

Investigations of the weathering of bituminous substances have been conducted by Hubbard and Reeve,[8] Church and Weiss,[9] Reeve and Anderton,[10] also Reeve and Lewis.[11] The changes brought about upon exposure to the elements are quite complicated, involving one or more of the following reactions:

Evaporation. This represents the gradual loss of volatile constituents on exposure to air and the sun's heat. Certain bituminous materials evaporate quite rapidly, and especially the tars. With any bituminous substance, the rate of evaporation depends almost entirely upon the temperature. Other things being equal, the higher the temperature the greater will be the volatilization. The determination of volatile matter (Test 16) is usually regarded to be an accelerated evaporation test, which is supposed to show in a relatively short time at an elevated temperature, what takes place over a lengthy period when exposed naturally to the air and sun. This is not, however, strictly correct, as will be explained below.

Oxidation. This takes place on exposure to air and progresses more rapidly at high than at low temperatures. The effect of oxidation is two-fold, and involves the direct union of oxygen with the bituminous substances, also the elimination of a portion of the hydrogen or carbon in the form of water or CO_2.[12] These reactions may be expressed roughly as follows:

$$C_xH_y+O=C_xH_yO$$
$$C_xH_y+O=C_xH_{y-2}+H_2O$$

also

$$C_xH_y+_2O=C_{x-1}H_y+CO_2.$$

The absorption of oxygen is accompanied by a *gain* in weight whereas the elimination of hydrogen or carbon is accompanied by a *loss* in weight. At low temperatures, these reactions are probably induced to a large extent by the actinic light rays.

A method has been proposed [13] for quantitatively measuring the volume of oxygen absorbed upon heating asphalts at 200° C. for 3 hours, the resultant changes in physical characteristics and components being recorded. One gram of the asphalt is intimately mixed with 25 g. of standard A.S.T.M. silica sand in a 50-ml. Erlenmeyer flask, which is heated in an oil-bath maintained at 200° C., while a flow of oxygen passes over the sample under a constant pressure of 2–3 mm. of xylene. The volume of oxygen absorbed is measured in ml. during 3 hours' heating. The water and CO_2 evolved are determined, likewise the loss or gain in weight of the sample. By this method, it has been found that the asphaltenes gain in weight during the reaction, whereas the asphaltic resins, petroleum resins and oils lose in weight. Moreover, asphaltenes from gilsonite and petroleum asphalts derived from U. S. petroleum oxidize less than the asphaltenes separated from Mexican petroleum asphalt.

It is recognizd that bituminous substances behave differently when heated in an inert atmosphere such as illuminating gas or nitrogen, than when heated under similar conditions in air or oxygen. In the former instance evaporation only takes place, whereas in the latter, evaporation occurs as before, but this at the same time is accompanied by a loss in weight due to elimination of hydrogen, also by a gain in weight caused by the absorption of oxygen. The extent and nature of these reactions will depend upon the substance itself, and also on the conditions to which it is subjected.

Carbonization. This represents the formation of "free carbon" in the bituminous material, and is induced by an extensive elimination of hydrogen, as indicated by the following reaction:

$$2C_xH_y+yO=2C_x+yH_2O.$$

In other words, it represents the elimination of hydrogen carried to an extreme. As a matter of fact, the deposit of free carbon generally contains a small percentage of hydrogen, and is rarely

composed of pure carbon. This reaction progresses most rapidly in sunlight, but will similarly take place upon subjecting the bituminous substance to a high temperature (see "Overheating").

Polymerization. This is due to a condensation or polymerization of the molecules, and manifests itself by a hardening or "setting" of the substance.[14] This polymerization has also been termed "thixotropy" or "spontaneous hardening" and is comparable, in a way, to the hardening or setting of Portland cement. It occurs usually to a greater extent on the surface of the material than it does beneath. The reaction may be expressed as follows:

$$nC_xH_y = C_{nx}H_{ny}$$

Bituminous materials after being freshly melted will appear softer and show a lower fusing-point than upon standing a day or two. For this reason it is recommended that the hardness and fusing-point be determined on the freshly melted material.

A method has been described [15] for quantitatively measuring the increase in viscosity of asphalts with time, by means of the Falling Coaxial Cylinder Method (Test 8*h*), the viscosity being expressed in absolute units (poises) at atmospheric temperatures. By plotting the log-viscosity against the time on a logarithmic scale, straight lines are obtained, the slope of which give quantitative measures of the rates of hardening. Assuming 100 hours as the basis for comparing various asphalts, the following expression results, which is termed the "Asphalt Aging Index":

$$\text{A.A.I.} = m$$

where m is the slope of the log-viscosity versus log-time curve. In this manner, it has been ascertained that the hardening is not caused by surface evaporation, and furthermore, that gentle heating of the samples brought their viscosities back almost to the original values.

Polymerization similarly takes place to a greater or lesser extent on heating bituminous materials to a high temperature, and is especially noticeable in fatty-acid pitches, some of which set and become infusible upon being heated in the neighborhood of 300° C., in the same manner as china-wood oil.

Effects of Moisture. All bituminous substances are more or less affected upon exposure to moisture, which manifests itself in two

ways, namely by the actual absorption of water and by the gradual leaching out of soluble constituents. These actions become intensified when the substance has oxidized, since oxygenated substances seem to have a greater affinity for moisture than the hydrocarbons themselves.

The moisture-absorbing properties of bituminous substances may be demonstrated optically by pasting a postage stamp on a piece of glass and coating it with a film of the bituminous substance applied in the form of paint. After the solvent has evaporated, the sheet of glass is immersed in water. Within twenty-four to forty-eight hours the water will be observed to have permeated the film, loosening the postage stamp, and forming a blister underneath.[16]

It has also been noted [17] that the bleaching of natural rock asphalts upon exposure to the weather is occasioned by the oxidation of the asphaltic constituents, which are thereupon readily leached out under the influence of atmospheric moisture. In addition, it has been found [18] that samples of asphalt, when placed in glass U-tubes in water in an atmosphere of oxygen and exposed to the action of an arc-light are gradually converted into water-soluble products containing acid- and ketone-bodies. Both light and oxygen are necessary factors for the formation of soluble products. These show an acid reaction and are precipitated by basic lead acetate.

The evaporation of volatile constituents upon aging leaves pores, which serve to increase the moisture-absorption, and at the same time permits free ingress of atmospheric oxygen. The latter serves to oxidize the bituminous constituents into water-soluble products, which in turn are leached out by the water, thereby increasing the porosity and augmenting the process of disintegration. The presence of clay or other associated mineral, animal, or vegetable constituents having an affinity for moisture, serves to hasten the destruction of bituminous substances. Animal and vegetable fibers associated with bituminized fabrics, in contact with moisture are likewise decomposed by the ravages of mildew and molds.

The following constitutes a résumé of the changes occasioned by the weathering process:

Gains:

(1) Oxidation products formed through the addition of oxygen.

Losses:
(2) Evaporation of the volatile constituents.
(3) Elimination of hydrogen (as H_2O) by oxidation.
(4) Elimination of carbon (as CO_2) by oxidation.
(5) Leaching out of soluble oxidation products (in item 1) by the action of water.

Alterations:
(6) Polymerization.
(7) Generation of free carbon.

It should be noted that there is more artificial hardening due to causes other than loss of volatile matter in actual weathering, than under the laboratory heat tests. There is no relation between the ductility of the residue in the laboratory heat test and the ductility of the substance in actual weathering. Substances showing carbenes will rapidly undergo carbonization under actual weathering, and the reverse is also true.[19]

Exposure to the weather affects the physical and chemical characteristics of bituminous substances in the following manner, viz.:

(Test 1)	Color in mass	Becomes lighter
(Test 2)	Homogeneity	Destroyed by the formation of free carbon
(Test 5)	Lustre	Disappears, the surface becoming dull
(Test 6)	Streak	Often changes from a black to a brown, and sometimes to a yellow
(Test 7)	Specific gravity	Increases
(Test 8)	Viscosity	Increases
(Test 9)	Hardness	Increases
(Test 10)	Ductility	Decreases
(Test 11)	Tensile strength	Decreases
(Test 12)	Adhesiveness	Decreases
(Test 13)	Breaking-point	Increases
(Test 14)	Solidifying-point	Increases
(Test 15)	Softening-point	Increases
(Test 15*g*)	Flow-point	Increases
(Test 15*h*)	Liquefying-point	Increases
(Test 15*i*)	Twisting-point	Increases
(Test 16)	Volatile matter	Decreases
(Test 17)	Flash-point	Increases
(Test 18)	Burning-point	Increases
(Test 19)	Fixed carbon	Increases
(Test 21)	Solubility in carbon disulfide	Decreases
	Non-mineral matter insoluble	Increases
(Test 22)	Carbenes	Variable
(Test 23)	Solubility in 88° petroleum naphtha	Decreases
(Test 24)	Free carbon	Increases
(Test 37*f*)	Unsaponifiable constituents	Unchanged
(Test 37*g*)	Saponifiable constituents	Unchanged
(Test 37*h*)	Glycerol	Unchanged

The weather-resisting properties of bituminous substances are of primary importance in the case of bituminized roof coverings, bituminous lacquers, cements, varnishes and enamels, on account of the relatively *thin* layers in which these products are customarily employed.

Two classes of weathering tests may be used, one of which consists in actually exposing the material to the effects of the elements, and the other in subjecting the material in the laboratory to the influence of artificial conditions of light, moisture and heat which duplicate as closely as possible the natural agencies, but in an intensified form. The latter has been aptly termed an "accelerated" weathering test.

Test 101. Actual Weathering Test. This test is usually slow, but the results are unquestionably reliable, since they duplicate actual service conditions.

(*a*) *Testing Bituminized Fabrics.* The following system has been adopted by the author for conducting exposure tests on bituminized roll-roofings, viz.: Sections 18 in. by 36 in., or 18 in. by 32 in., depending upon whether the roofing is 36 or 32 in. wide, are taken across the sheet, the cutting being sharp and square. These are exposed on a platform, composed of ⅞-in. tongued and grooved boards, preferably pine, having a 2-in. slope to the south, the samples being nailed with large-headed galvanized barbed roofing nails at the four corners, midway across the 18-in. edges, and at three intermediate points along the 26- or 32-in. edges, a total of 12 nails being used.

Both the indoor and exposed samples are examined at the following intervals, viz.: one-half year, one, two, three, four, five and ten years, and the data recorded relative to the appearance and condition of the specimens.

The eight specimens illustrated in Fig. 322 represent the typical surface conditions of smooth-surfaced roll-roofings after exposure to the weather for a period of 5 years. Specimen *A* is unchanged and homogeneous, specimen *B* is covered with fine checks, specimen *C* is covered with coarse checks, in specimen *D* the checks are disappearing (i.e., they were in evidence at the time of the previous observation, but have since largely disappeared), specimen *E* shows a few blisters, specimen *F* is covered with hair cracks, specimen *G*

is covered with coarse cracks, and in specimen *H* the felt is largely exposed. Figure 323 shows specimens *A* and *H* enlarged 3½ diameters. The veined surface of *A* shows up very distinctly, and also the characteristic uneven appearance of the roofing when the weather-coating has worn off and the felt fibers exposed, as in *H*.

"Checking" is distinctly a surface phenomenon which manifests itself with certain substances on exposure. The checks rarely extend entirely through the bituminous coating, and are seemingly caused by the hardening and contraction of the upper stratum (i.e., shrinkage of the skin), resulting in a tension which is sufficient to cause it to crack and slide over the softer sub-stratum. One theory is that checking or "grooving" is caused by the difference in surface-tension between the oily constituents and the asphaltenes, under the influence of light. Bituminous substances which are largely influenced by changes in temperature (in other words having a high susceptibility index) are likely to check. As the "spontaneous hardening" progresses downward into the lower layers, the checks gradually disappear. It has been suggested that the contraction and checking of asphalts may be obviated by adding up to 10 per cent by weight of coal-tar pitch, or 2 to 3 per cent by weight of anti-oxidants, such as are used in the rubber industry. Antioxidants are more correctly termed "antioxygens." There are three general classes: (1) amines, (2) phenols, and (3) aldehyde-amine condensation products. The antioxygens that have been proposed for use with bituminous substances include: phenyl-beta-naphthylamine, phenyl-alpha-naphthylamine, hydroquinone, cachetol, pyrocachetol, guaiacol, dipentene, pyridine, quinoline, aniline, dimethylaniline,[20] alkaloids (e.g., nicotine),[21] phenol-formaldehyde resin,[22] titanium phthalate,[23] nitrogenous bases (e.g., bone tar).[24]

The incorporation of finely-divided fillers will tend: (a) to prevent checking and cracking; (b) to improve the weather-resistance, by protecting the coating from the disintegrative action of sunlight; (c) to give the coating "body," so that it will not flow so readily; (d) to increase its "plastic range" (i.e., by increasing its fusing-point and at the same time decreasing its solidifying-point); and (e) to improve its resistance to mechanical attrition. The optimum characteristics of such fillers are: (a) they shall be

finely-divided; (b) they shall be light-excluding; (c) they shall be of themselves chemically inert and resistant to weathering influences (i.e., heat, moisture, acids, etc.); (d) the mixing with the bituminous constituents shall be thorough and complete, so that the mixture will be homogeneous; (e) the filler must be used in suitable proportions; and (f) the use of two fillers will often give better results than the use of either component alone in the same total quantity. Different fillers act differently with any one asphalt. Similarly, each asphalt behaves differently with any particular filler. Hence, every asphalt-filler combination must be tested out individually, to arrive at its suitability. The properties of combinations involving the use of two or more fillers are not always additive. The fusing-point of asphalts is increased by the addition of fillers, and the finer the subdivision of the filler, the greater will be the increase. When a coarse and a fine filler are added together, the fusing-point of the mixture will be increased more than would be the case if the total quantity of fine filler alone were added.

"Blistering" is caused by one or more of the following factors, viz.:

(*a*) Temperature: If the saturated sheet is at too high a temperature (i.e., above 375° F. in the case of asphaltic saturants) when the coating is applied, or if the coating itself is applied too hot, then non-condensable gaseous vapors are apt to be imprisoned in the bituminous constituents, which under the heat of the sun will expand and form unsightly blisters.

(*b*) Entrapped air: This is one of the principal causes of blistering. The air may be entrapped in the coating, because of the prior blowing process, or as a result of incorporating fillers, or in the mechanical operation of applying the melted coating to the sheet of saturated felt.

(*c*) Volatile matter: Saturants, and to a lesser extent coatings, which carry an abnormally large proportion of volatile constituents are likely to blister, since these have a tendency to vaporize when subjected to the sun's heat and the resulting pressure can only be relieved in this manner.[25]

(*d*) Fusing-point of the coating: Coatings having high fusing-points are less apt to blister than those made of a low fusing-point,

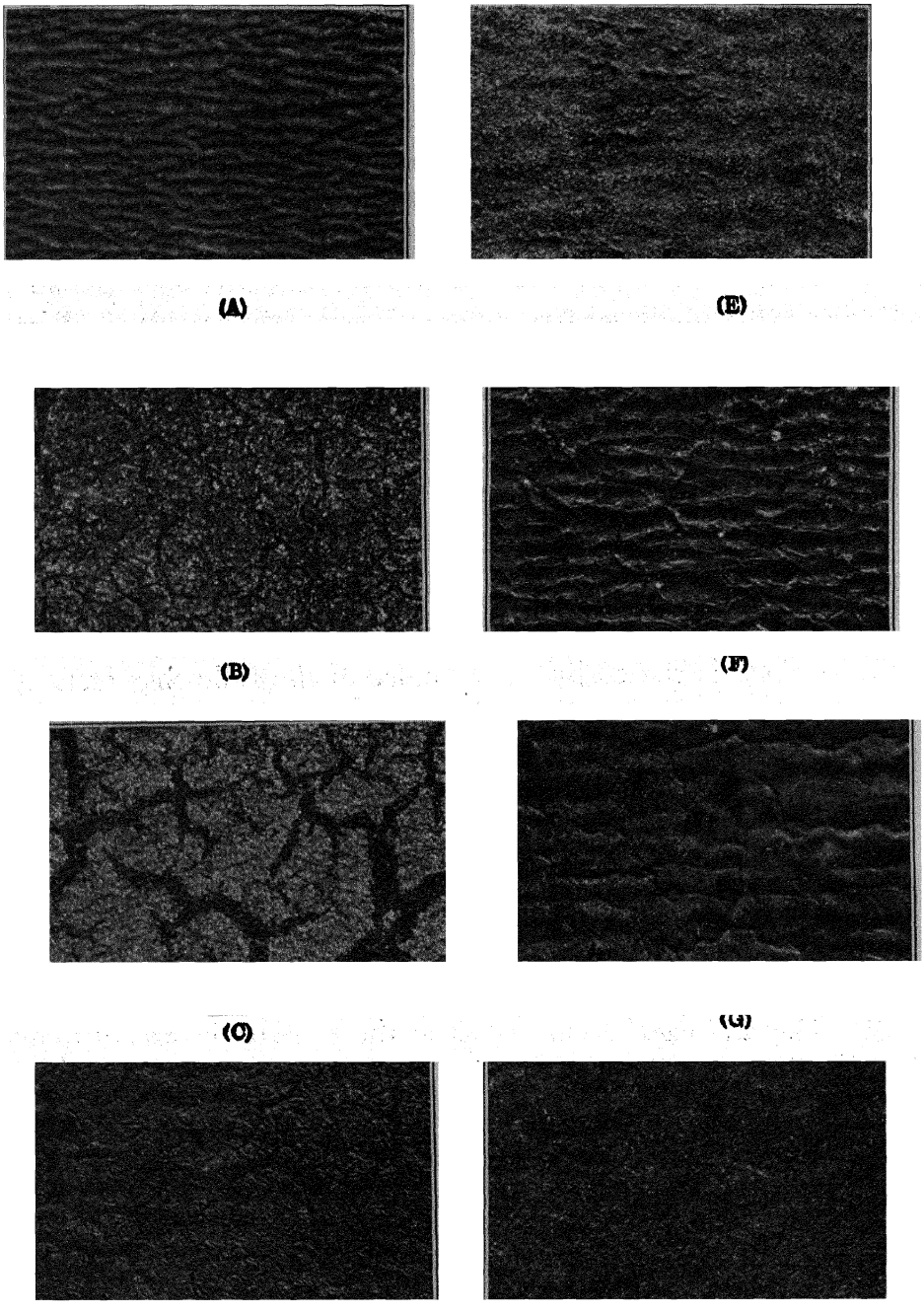
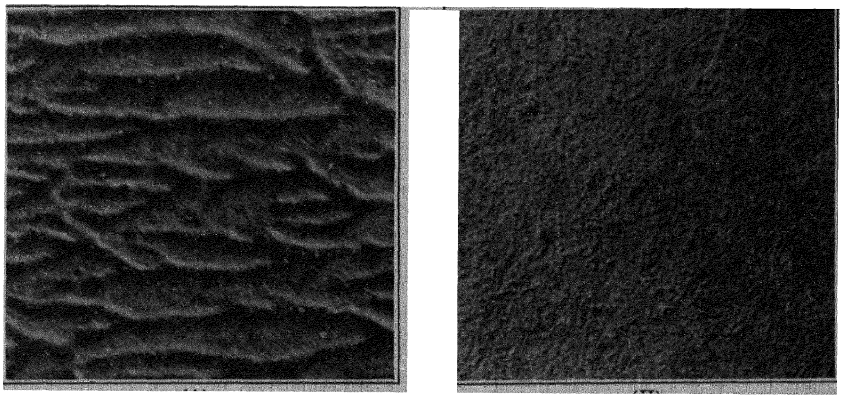


FIG. 322.—Effects of Exposure on Smooth-surfaced Prepared Roofings.

other things being equal. The inclusion of a certain amount of suitable filler in low fusing-point coatings will also tend to overcome this trouble.

(*e*) Character of the surfacing: In the case of mineral-surfaced roofings and shingles, the porosity of the slate or other mineral surfacing proves a contributing factor, due to the likelihood of porous minerals absorbing moisture and conducting it to the interior of the sheet. Moisture imprisoned in this manner will suddenly expand under the heat of the sun, and by following the path of least resistance, will distort the coating and form pockets or blisters.



(A) (H)

FIG. 323.—Enlargements of Specimens A and H in Fig. 322.

Hair cracks are caused by the contraction of the bituminous material, and take place with substances which are hard, brittle, and devoid of elastic properties. The action is aggravated by the use of soft, plastic saturating materials in conjunction with a hard and brittle coating. The cracks usually extend all the way through the bituminous coating, and will neither seal up nor disappear in time, as is the case with the checks.

The predominating color is a criterion of the rapidity with which the soapstone or mineral matter on the surface disappears, and "dusting" furnishes an indication of the rate with which the bituminous coating weathers away on exposure. As bituminous substances weather, they form a pulverulent chalk-like mass having but little coherence, and which is therefore easily removed by wiping

with a cloth. This corresponds to "chalking" of linseed-oil paint films. The influence of rubbing on the color is of supplemental value, furnishing an indication of how deep the weathering has progressed.

The pliability of the roofing shows to what extent the roofing has "dried out," bearing in mind that when the pliability decreases to a certain extent, the sheet can no longer fulfil its function properly, but will break upon being subjected to severe vibratory strains.

The tensile strength indicates the extent to which the weathering has weakened the roofing, also an approximation of its residual wearing qualities. By constructing a curve of the tensile strength of the sheet at different intervals, some idea may be gained of its probable durability. As the roofing ages indoors, it gradually gains in strength, until it finally remains constant. A duplicate specimen exposed outdoors, rapidy gains in strength up to a certain point, which corresponds to the disappearance of its weather coating. The tensile strength will thereupon decrease until it falls below the corresponding strength of the indoor sample. The roofing reaches its "mean effective life" when the strength curve of the outdoor sample crosses the curve of the indoor sample. This will be made clear by Fig. 324, showing the strength curves of representative high-grade light, medium and heavy weight smooth-surfaced prepared roofings weighing 32, 42 and 52 lb. net per 108 sq. ft. The solid lines represent the strength of the outdoor samples, and the dotted lines the corresponding strength of samples aged indoors. The figures in Table CXVI will interpret the diagram.

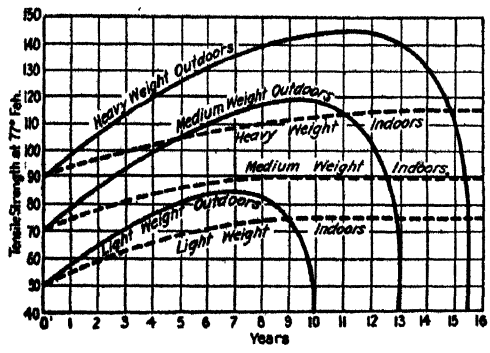


FIG. 324.—Tensile Strength Curves of Prepared Roofings on Exposure.

The mean effective lives of the roofings in question may be taken as nine, twelve and one-half and fifteen years respectively, and the maximum effective lives as ten, thirteen and fifteen and one-half years.[26]

(*b*) *Testing Bituminous-solvent Compositions.* These may

TABLE CXVI

EFFECT OF ACTUAL WEATHERING ON THE STRENGTH OF PREPARED-ROOFING

	Light Weight (35 lb. per 100 sq. ft.)		Medium Weight (45 lb. per 100 sq. ft.)		Heavy Weight (55 lb. per 100 sq. ft.)	
	Years	Lbs.	Years	Lbs.	Years	Lbs.
Original strength	0	50	0	70	0	90
Maximum strength outdoor sample	7	85	10	120	12	145
Outdoor sample same strength as indoor	9	75	12½	90	15	115
Outdoor sample same strength as originally	10	50	13	70	15½	90

similarly be tested by applying them in one or more coats to steel sheets or wooden panels, and observing their appearance at regular intervals. The following features should be recorded:

(1) Loss of lustre.
(2) Condition of the exposed surface.
(3) Amount of dusting.
(4) Influence of rubbing on the color.
(5) Any chipping of the composition and exposure of the underlying surface.
(6) Any corrosion, in the case of the steel plates.

Much has been written concerning the methods for performing exposure tests on paints, including bituminous coatings and dispersions, and for further information on this subject, the reader is referred elsewhere.[27]

(*c*) *Testing Crude, Refined or Blended Bituminous Substances.* This test is performed by melting the bituminous substance at the lowest possible temperature in a shallow, flat-bottomed glass dish in a layer 0.025 in. thick. A so-called Petri dish may conveniently be used for this purpose, measuring approximately 3 in. in diameter and ½ in. deep, thereby exposing a surface of approximately 7 sq. in. (451.6 sq. mm.). This will require 3 ml. of the material, which, multiplied by its specific gravity, will give the corresponding weight in grams. The proper weight of bituminous substance is introduced into the dish and then melted in an air-bath at a temperature slightly above its fusing-point, so as not to result in any appreciable loss of volatile constituents. An alternate procedure consists in

applying a layer of the bituminous material 0.025 in. thick to a sheet of thin aluminium, 3 by 6 in., and 0.050 to 0.065 in. thick. This may be conveniently done by melting the material in a layer somewhat thicker, then placing the specimen in a mold of the proper depth (i.e., the thickness of the metal sheet, plus 0.025 in.), and finally rolling it cold between a pair of heated metal rollers until all but 0.025 in. ± 0.003 in. of the bituminous coating is removed. The dishes or panels are exposed to the elements in a dustproof box having a quartz glass top, so as to permit access of the largest amount of ultra-violet rays. Air circulation is provided by means of side vents covered with cotton gauze and the specimens are protected from rain by overhanging eaves. The specimens are examined at the end of six months and one year, and the following observations recorded:

(*a*) Appearance to the eye and under the microscope.
(*b*) Amount of dusting and influence of rubbing on the color.
(*c*) Increase in softening-point over that of the original material. (Test 15.)
(*d*) Comparison of the breaking-point (Test 13) with that of the original material (where the test is made on sheet metal).
(*e*) Increase of the amount of material insoluble in carbon disulfide over that originally present. (Test 21.)

Figure 325 illustrates the appearance of typical specimens after having been exposed for one year. In the case of asphalts, the fusing-point increased up to 150° F. over the original fusing-point, and the material insoluble in carbon disulfide showed an increase ranging from 1 to 10 per cent over the original figures.

Tests have been conducted [28] to ascertain the effects of weathering on the asphaltic constituents separated in accordance with Test 38. The results obtained from Mexican, German and Russian residual asphalts are given in Table CXVII. It will be observed that the oily constituents progressively decrease, whereas the asphaltic resins and the hard asphalt in the asphaltenes are correspondingly increased.

Tests involving exposure in very thin films have also been described.[29]

Test 102. Accelerated Weathering Test. The apparatus developed for this purpose has been developed by the Bureau of Stand-

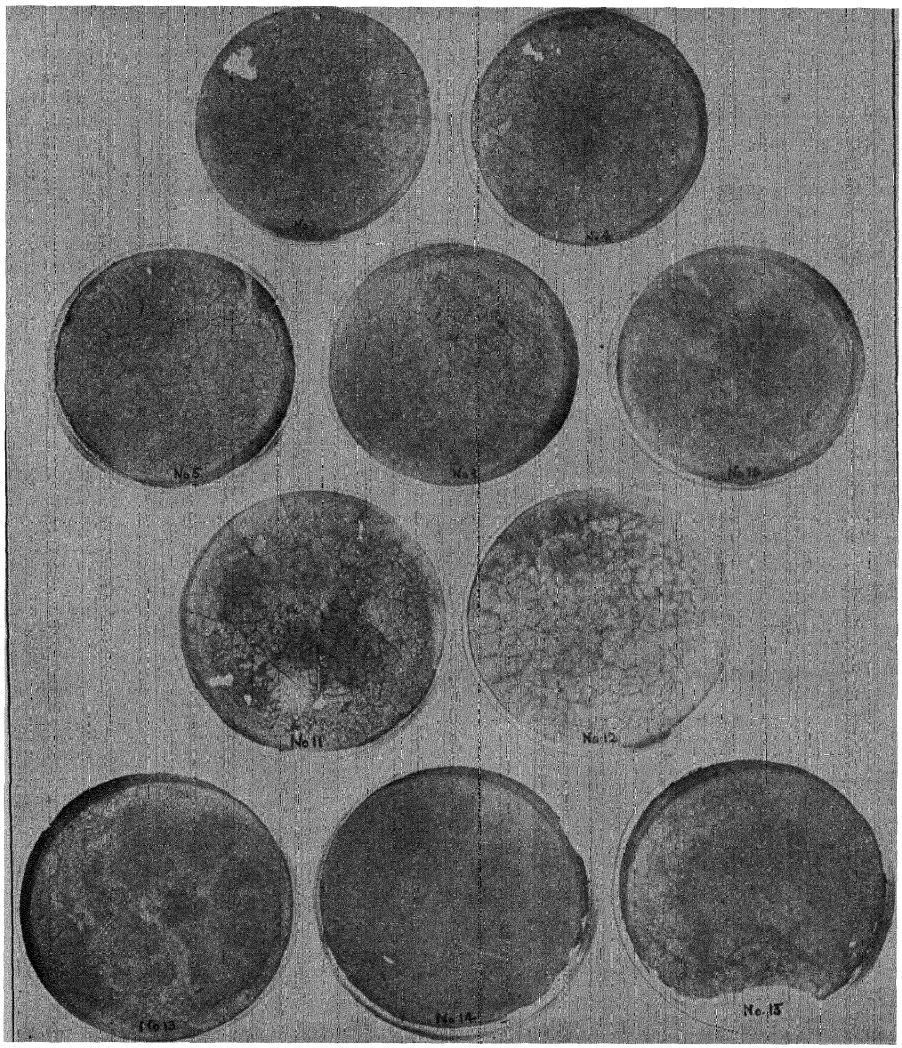


FIG. 325.—Typical Bituminous Substances after One Year's Exposure to the Weather.

ards, Washington D. C.[30] The following procedure has been proposed for testing bituminous substances:

TABLE CXVII

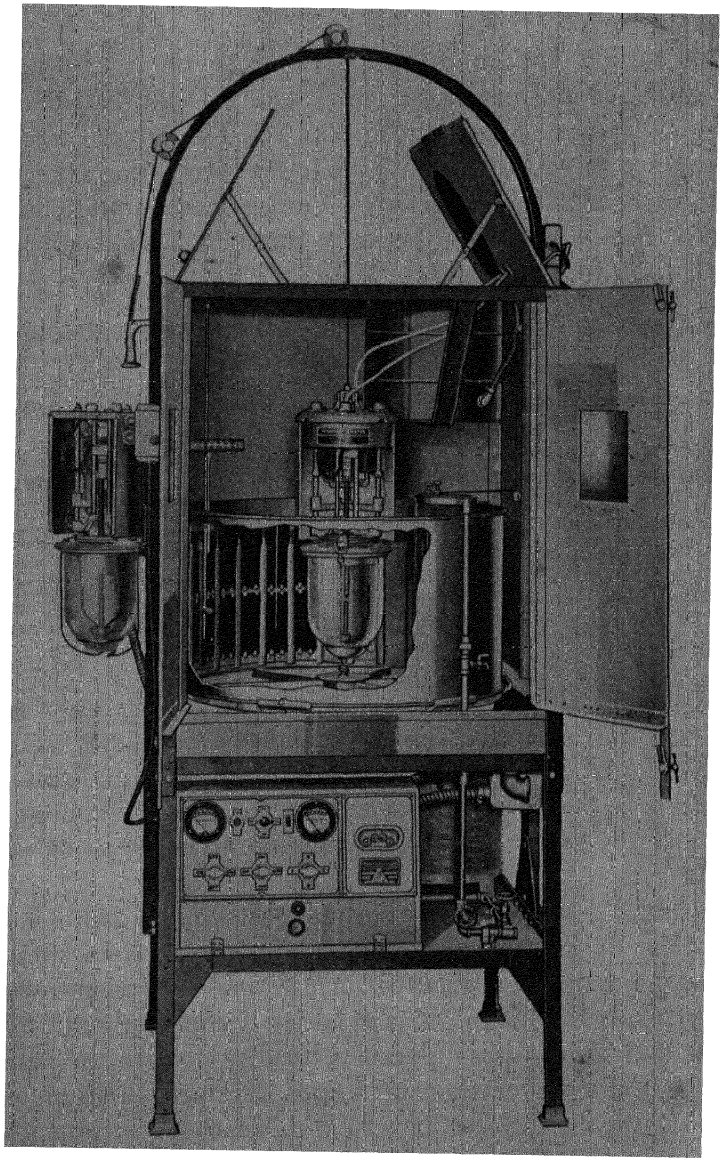
EFFECT OF ACTUAL WEATHERING ON THE CHARACTERISTICS OF PETROLEUM ASPHALTS

	Mexican Residual Asphalt	German Residual Asphalt	Russian Residual Asphalt
Original Material:			
Fusing-point (K. and S.)	24.8° C.	28.1° C.	30.7° C.
Dropping-point (Ubbelohde)	50.8° C.	50.3° C.	50.8° C.
Oily constituents	49.29%	39.11%	46.67%
Asphalt resins	24.76%	43.75%	32.17%
Resins in asphaltenes	21.34%	16.02%	20.55%
Hard asphalt in asphaltenes	4.61%	1.12%	0.61%
Total	100.00%	100.00%	100.00%
After Exposure in Dark to Air Indoors 5 Mos. (layer ¼–½ mm.):			
Fusing-point (K. and S.)	+ 6.4° C.	+ 3.3° C.	+ 0.1° C.
Dropping-point (Ubbelohde)	+ 3.2° C.	+ 4.7° C.	+ 1.2° C.
Oily constituents	− 9.33%	− 2.39%	− 1.03%
Asphalt resins	+14.77%	− 0.50%	+ 1.51%
Resins in asphaltenes	− 5.56%	+ 2.66%	− 0.62%
Hard asphalt in asphaltenes	+ 0.12%	+ 0.23%	+ 0.14%
After Exposure to Weather Outdoors 5 Mos. (layer ¼–½ mm.):			
Fusing-point (K. and S.)	+41.4° C.	+29.3° C.	+25.2° C.
Dropping-point (Ubbelohde)	+39.5° C.	+29.7° C.	+26.7° C.
Oily constituents	−20.75%	−13.59%	− 8.43%
Asphalt resins	+ 5.89%	− 8.57%	− 9.42%
Resins in asphaltenes	+13.11%	+18.40%	+13.03%
Hard asphalt in asphaltenes	+ 1.75%	+ 3.76%	+ 4.82%

This method is intended to determine the durability of bituminous materials by producing rapid deterioration of the materials under conditions simulating extreme outdoor exposure.

The apparatus required consists of the actual weathering equipment, a trimmer to prepare the bituminous coatings and an instrument to determine the extent of the "weathering," as follows:

Accelerated Weathering Equipment. The actual weathering equipment shall consist of a carbon arc lamp, a cylinder, a sprinkler, a spray, and an arrangement for temperature control during the light period. Fig. 326 shows a complete unit suitable for testing the materials in the vertical position. Soft materials are tested



Courtesy Bureau of Standards.

FIG. 326.—Accelerated Weathering Apparatus for Panels in the Vertical Position.

in the horizontal position, using one or two lamps as shown in Fig. 327.

The carbon arc lamps * shall operate on a 220-volt line, either direct or alternating current. The voltage shall be reduced by means of resistances to 130 to 145 v. The direct current lamp shall operate at 12 to 14 amps., while the alternating current lamp shall operate at 15 to 17 amps. Under these conditions the intensity of the light (15 in. opposite the flame) shall approximate 3500 to 4000 foot-candles (see Note).

NOTE. The lamp shall have approximately the following spectral range, expressed in percentage of total radiation to limit of fluorite transmission in infra-red (12,000 μμ): 279-290 μμ: 0.8 per cent; 290-320 μμ: 4.1 per cent; 320-360 μμ: 6.0 per cent; 360-480 μμ: 14.5 per cent; 480-600 μμ: 8.0 per cent; 600-1400 μμ: 14.8 per cent; 1400-4200 μμ: 21.4 per cent; and 4200-12,000 μμ: 30.4 per cent; total: 100.0 per cent.

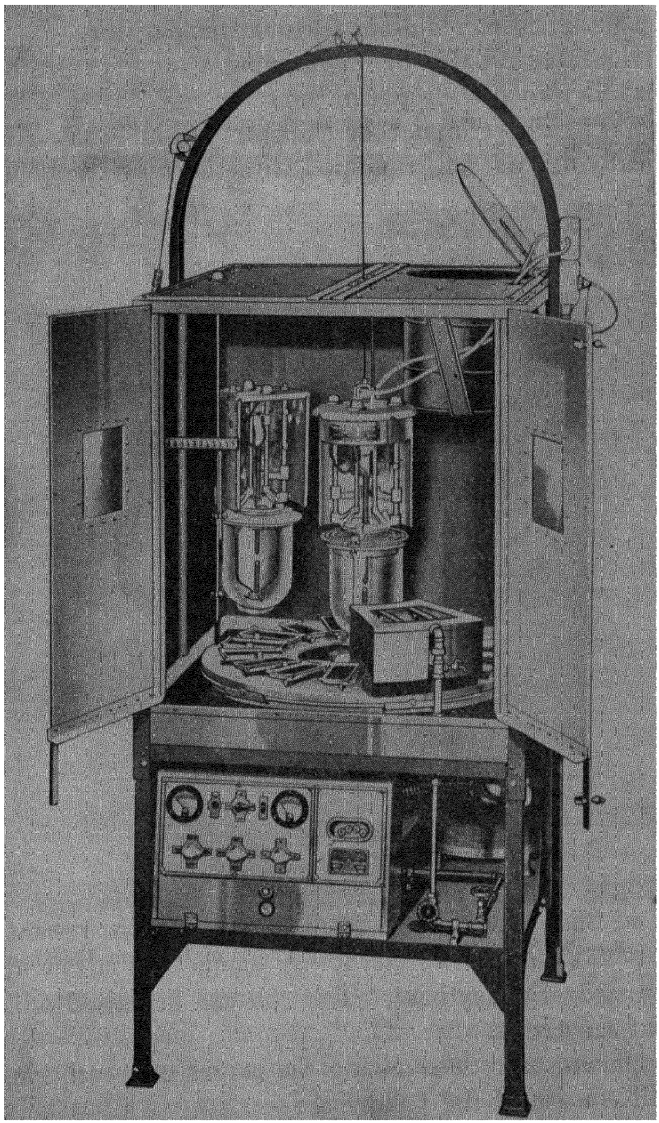
It is advisable to check the strength of the light from time to time as follows: A freshly prepared solution of uranoxalate is made by adding 4.2 g. of uranium sulfate to 1 liter of N/10 oxalic acid solution. Two quartz tubes, each containing 25 ml. of the uranoxalate solution are supported 6 in. from the arc, one on each side, and exposed to the source of light for 30 minutes. The solutions are then acidified with sulfuric acid and titrated with N/10 $KMnO_4$ solution at 70° C. The mean of the two readings is taken. The intensity of the arc is measured by the loss in strength of the uranoxalate solution. The original actinic strength of the arc is usually between 40 and 50 per cent, and should not be allowed to fall below 30-35 per cent.

The lamp shall be equipped with an ammeter, a watt-hour meter and a voltmeter, all three instruments being connected across the arc. Solid carbon electrodes shall be used in the direct-current lamp, while in the alternating-current lamp one solid and one cored carbon electrode shall be used. The lamp globes shall be made of Corex-D or some similar glass.

A cylinder 30 in. in diameter and about 15 in. in depth, equipped with slots or hooks to hold the test specimens and connected with a revolving mechanism, geared to furnish one revolution in 20 minutes, surrounds the lamp. The cylinder is equipped with a rotary sprinkler and with a spray placed vertically, close to the inner rim. The lamp and cylinder shall be suspended over a pan about 4 to 5 in. in depth, the latter equipped with an overflow pipe.

For maintaining a uniform temperature during the light period the weathering apparatus shall be equipped with a thermo-regulator and ventilating equipment which permit automatic maintenance of

* These requirements are for the lamps now in general use. Other lamps will probably have other requirements.



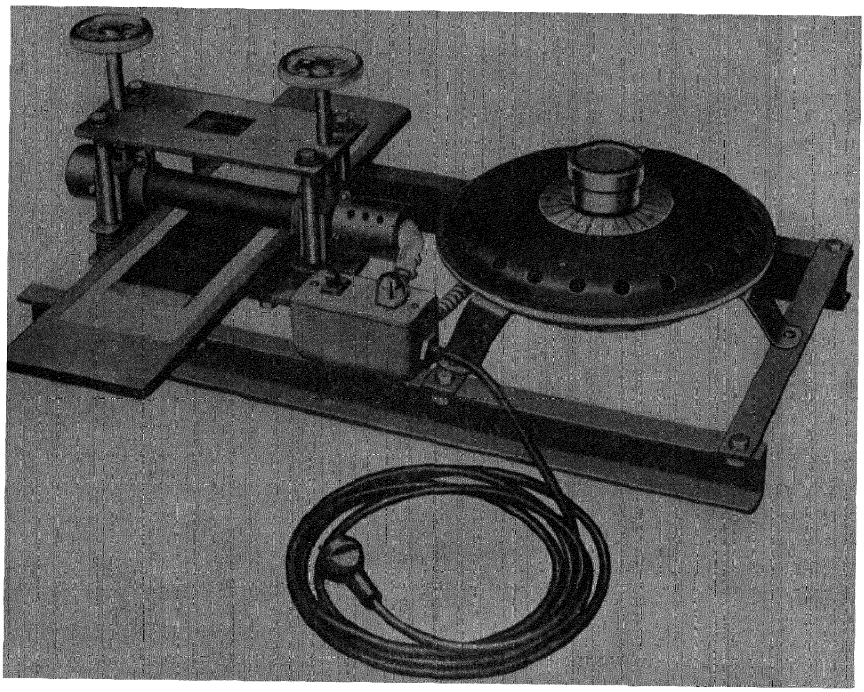
Courtesy Bureau of Standards

FIG. 327.—Accelerated Weathering Apparatus for Testing Soft Materials in the Horizontal Position.

temperature, or the accelerated weathering machine shall be placed in a room designed to maintain uniform temperature.

A refrigerator capable of maintaining a temperature of — 10° F. (— 23° C.) forms a separate unit.

Trimmer. The trimmer or "doctor" (Fig. 328) consists of an electrically-heated roll, equipped with thermostatic control, sus-



Courtesy Bureau of Standards

FIG. 328.—The Trimmer for Preparing Panels of Uniform Thickness.

pended over a stationary base. The roll can be raised or lowered by means of set-screws.

The apparatus for determining the extent of weathering (Fig. 328*a*) shall consist of the following:

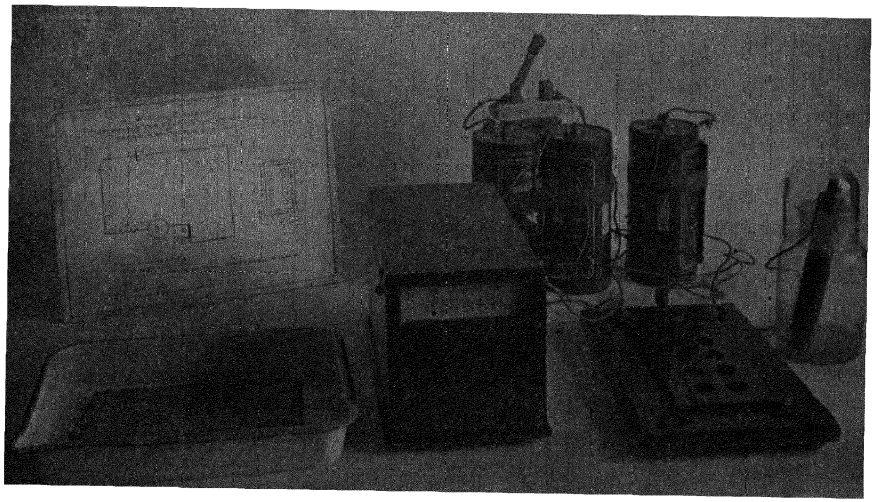
One terminal of a single 1.4 v. dry cell, *A*, shall be connected to a 1-megohm fixed resistance, *B*, thence to a galvanometer, *G*, and thence to a cell, *D*, containing a dilute electrolyte. The cell, *D*, shall be made of a copper tube fitted into a piece of bakelite tubing which acts as an insulator. A piece of maplewood dowel

pin shall fit snugly into the copper tube. This wood pin acts as a "wick" to carry the electrolyte (a 0.5 per cent solution of magnesium sulfate). Copper gage contained in the wood frame, *E*, shall be connected to the other terminal of the dry cell, *A*. A bakelite template, *P*, with ten holes about 1.6 cm. in diameter equally spaced over the panel definitely places each individual test.

Method of Preparing Test Panels.

(*a*) Bituminized Fabrics are simply cut to the proper size.

(*b*) Bituminous-Solvent Compositions may be applied to glass



Courtesy Bureau of Standards

FIG. 328a.—Conductivity Test Apparatus.

or aluminium panels in two or three coats, and should be tested at least in duplicate, to compensate for any irregularities in application.

(*c*) Bituminous Compositions should be applied to aluminium panels in the following manner:[81] The sample shall be melted at the lowest possible temperature in a seamless can and then poured upon the heated (130 to 160° F., 55 to 70° C.) 3 by 6 by 1/16 in. aluminium panel. Just prior to pouring, the aluminium panel shall be placed on a piece of folded typewriter paper on the base of the trimmer. The paper takes up excess bituminous material and keeps the base clean. For convenience it is better to use two such papers so that one remains stationary and the other draws the panel under

the trimmer, Fig. 328. The panel and the bituminous material shall then be drawn under the previously set and heated trimmer so that a bituminous film of 0.025 in. ± 0.003 in. is obtained. It may be necessary to draw the panel back and forth under the heated roll several times. The panel shall then be allowed to cool, the edges shall be cleaned and then calipered, five readings being taken lengthwise. It is important in all these operations that overheating of the bituminous material be avoided.

After each coating operation, the trimmer shall be wiped with cloth or paper to keep the roll clean and smooth. The temperature of the trimmer is approximately correct when paper held against it slightly browns.

Test Procedure. The test specimens shall be subjected to a daily weathering cycle consisting of periods of exposure to cold, light, and water, the weathering cycle being as follows:

Rain exposure for a period of ¼ hr.
Cold exposure for a period of 1¾ hr.
Light-spray for a period of 4 hr.
Rain exposure for a period of ½ hr.
Light exposure for a period of 17 hr.

For the light, rain, and light-spray exposure periods, the specimens shall be placed in the holders of the cylinder. For the rain period the lamp is raised above the cylinder, the latter covered, and the specimens are vigorously sprinkled with water by means of a rotary spray which operates at the bottom of the cylinder.

For the cold exposure period the specimens shall be placed in the refrigerator, previously cooled to — 10° F. (— 23° C.). Fifteen minutes are allowed to change panels from the cylinder to the refrigerator and the same time is again allowed after the cold period to return the specimens to the cylinder for the light-spray period.

For the light period the lamp is operated together with the revolving cylinder. During the light-spray period the light from the lamp shines on the specimens continuously while the water, running constantly from the spray at a fixed point, wets the specimens in the revolving cylinder once every 20 minutes.

The temperature at the surfaces of the specimens during the light period shall be 135 to 145° F. (57 to 63° C.). The tem-

perature shall be measured by placing a black bulb thermometer horizontally opposite the arc at the surface of the panels, or by means of a thermo-element. In the case of the light-spray period the specimens will, of course, be cooled as they pass by the water spray; the specimens should, however, warm to about 140° F. (60° C.) before being wet again. In cold weather, it may become necessary to use warm water for spraying in order to reach such a temperature. Water (2 to 4 in. in depth) shall be kept in the copper tank or pan all the time, the excess water flowing out over the overflow. The water modifies the temperature and humidifies the air during the light period.

The lamp globes shall be wash daily, and dried thoroughly.

The electrodes shall be changed daily. At this time the metal parts of the lamp (those inclosed by the glass globe) shall be cleaned by brushing, thus removing soot and rust.

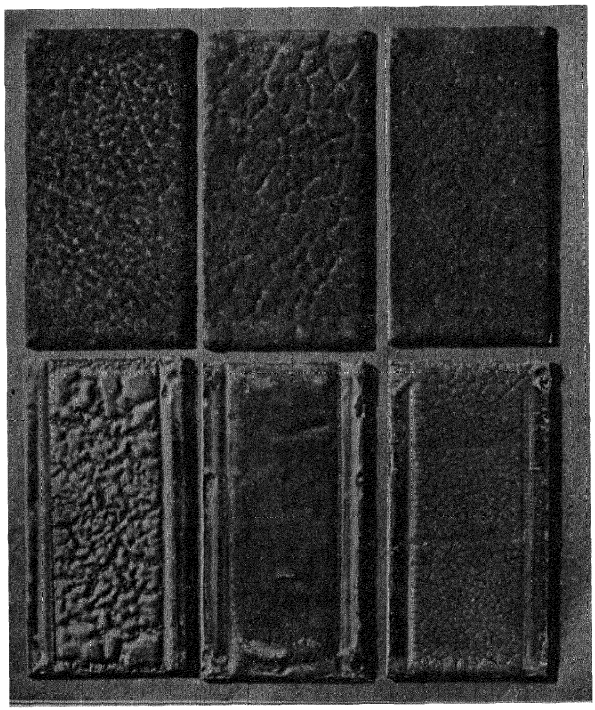
Determining Extent of Weathering. The results of accelerated tests are compared with the materials before weathering and the normal tests such as loss in pliability, tensile strength, loss in weight, solubility in standard solvents, etc., are carried out in accordance with known methods.

The principal test is the estimation of the surface differences before and after weathering. This may be carried out on instruments of the photo-electric type. All these tests are general but for bituminous materials surfaced on aluminium panels, however, the "conductivity test" (Fig. 328*a*) for determining the extent of weathering shall be made as follows:[80]

The panel shall be placed on the test surface, *E*, the aluminium side against the copper gauze, then covered with the bakelite template, *F*, and this shall be firmly clamped to the frame by spring clamps, thus pressing the panel firmly against the gauze. The wick, *D*, moistened with the electrolyte shall be brought in contact with the bituminous surface through the holes in the template, *F*. In order to insure rapid penetration a small quantity of alcohol may be added to the electrolyte. Some of the electrolyte shall be poured into the copper tube and when not in use the outer end of the wood plug shall be placed in a shallow layer of the same solution so as to keep the wood wet with the electrolyte. If cracks are present in the coating, the electrolyte will penetrate to the alu-

minium, closing the circuit and causing the galvanometer needle, *G*, to fluctuate. The conductivity test, when made, shall be made immediately following the completion of a light period. Any degree of fluctuation of the galvanometer, *G*, shall be considered positive.

The weathering test shall be considered completed when six or more positive tests (out of a possible ten) are obtained. The test



Courtesy Bureau of Standards.

FIG. 329.—Weathering of Typical Asphalts. Upper row: exposed one year out-of-doors Lower row: shows the same asphalts after being subjected to the weathering cycle for 70 days.

shall be reported as the number of weathering cycles necessary to obtain this result. For permanent records the test specimens shall be photographed. A Leica or a fingerprint camera is suitable for this purpose.

It has been proposed that in the case of bituminous-solvent compositions, the exposed specimen be bent double in a predetermined

time at a prescribed temperature over a rod of a fixed diameter. It is contended that the more durable the composition, the longer it may be exposed to the ultra-violet light before it will crack on bending.[32]

Figure 329 illustrates the appearance of typical asphalts, all having a fusing-point of 220° F. (R. and B. method), the upper row having been exposed out-of-doors for one year under quartz glass (Test 101*c*), and the lower row, representing the identical asphalts, after having been subjected to the accelerated weathering cycle (Test 102*c*) for 70 cycles.

The progress of the accelerated test may be followed by means of Test 38. Thus, a blown petroleum asphalt having a fusing-point of 220° F. behaved as follows:

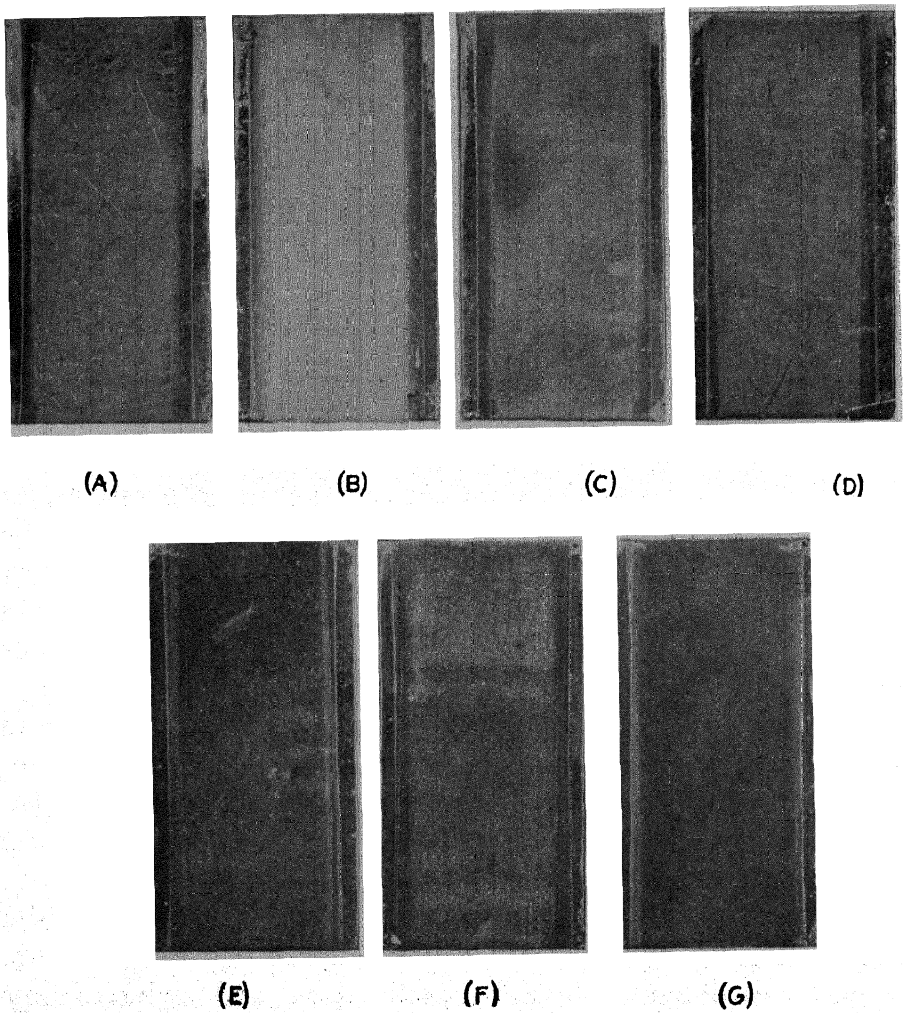
Cycles	Asphaltenes, %	Asphaltic Resins, %	Oily Constituents, %
Originally.....	26.2	4.8	68.0
After 21 cycles.....	34.6	1.2	61.4
After 80 cycles.....	40.6	1.2	56.6

It will be noted that the asphaltenes increase progressively at the expense of the oily constituents and to a lesser extent at the expense of the asphaltic resins. This is corroborated by heating at 200° C. in an atmosphere of oxygen for 3 hours. Asphaltenes became 96 per cent insoluble in carbon disulfide and asphaltic resins produced a product which was 31 per cent insoluble.

Table CXVIII shows the behavior of blown asphalts, with and without the addition of mineral fillers to the accelerated exposure test, the final appearances being illustrated in Fig. 330. Of the unfilled asphalts tested, Mid-continental stood first, Californian and Colombian second, and Texas third, in the order of their respective weather-resistance. Of the fillers tested, alberene flour (fine-mesh) stood first, slate flour stood second, talc third, limestone flour fourth, silica dust and dolomite flour fifth, silica sand and hydrated lime sixth. With any particular filler, the finer the mesh, the better proved to be the resistance.

Investigations have also been made [33] to show the effect of mineral fillers on the durability of asphalt coatings by exposing samples outdoors and to the accelerated weathering cycle. The same type of asphalt, made from the same crude in a single operation, but differing only in the duration of the blowing, was prepared with

softening-points (R. and B. method) of 194° and 225° F. respectively. These two asphalts were so blended that, when combined with the stipulated weight of filler, the softening-point of the resulting mixture was 225° F. The compositions and physical prop-



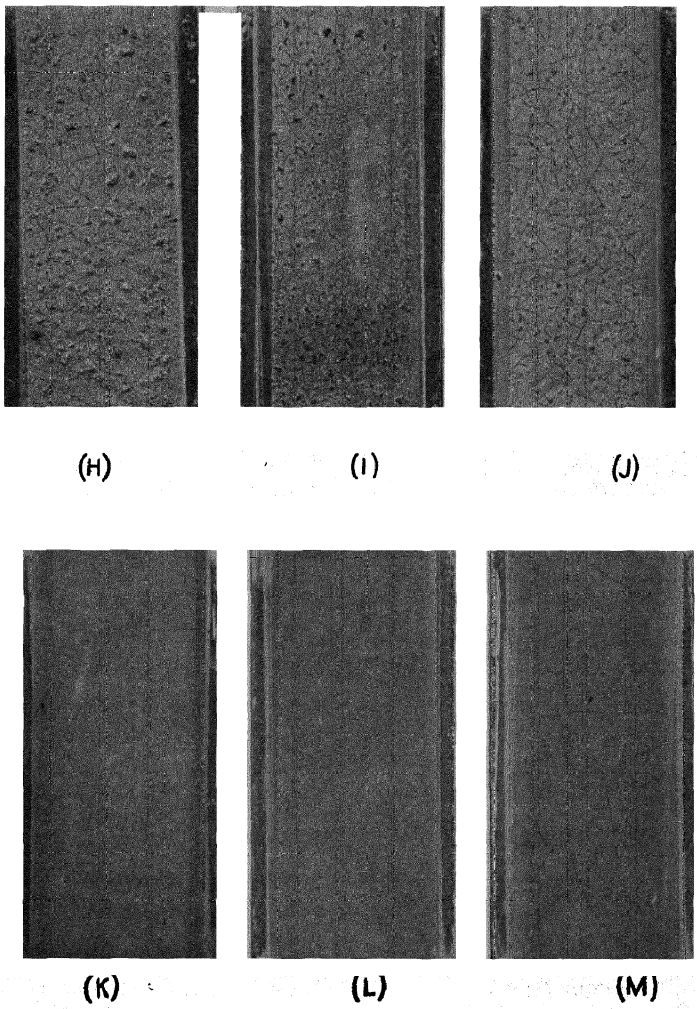
Courtesy Bureau of Standards

FIG. 330.—Accelerated Weathering of Blown Petroleum Asphalts.

erties of the various compositions tested are shown in Table CXIX. The physical properties of the fillers themselves are given in Table CXX, in which the "compacting weight" represents the weight of the filler per milliliter when thoroughly compacted by tapping, and the "fineness factor" ("*f*") is calculated by subtracting the com-

pacting weight from the density and dividing the result by the compacting weight.

The outdoor panels were exposed to the weather for 1½ years, whereupon they were examined for cracks visually and under a



Courtesy Bureau of Standards

FIG. 300.—Accelerated We ıering of Blown Petroleum Asphalts (Continued).

magnificatoin of ×12. Duplicate panels were subjected to the standard accelerated weathering test and the durability of the respective coatings expressed in cycles. It will be observed that in general the coatings failed in the same order in the outdoor tests

TABLE CXVIII

ACCELERATED WEATHERING TESTS—BLOWN PETROLEUM ASPHALTS

Figure 330	Blown Asphalt R. and B. Fusing-pt. °F.	Mineral Filler Added (35% by Weight)	No Cracks Visible to the Eye	Cracks Visible; Aluminium Panel Not Exposed	Cracked through; Aluminium Panel Visible (= Failure)
Produced from California Petroleum:			*Cycles*	*Cycles*	*Cycles*
(A)	221	None	Below 40	→	At 41–42
(B)	221	Alberene flour—350 mesh	Below 101 (*)	.....	....
...	221	" " —200–300 mesh.	Below 88	→	At 93(†)
...	221	" " —150–200 mesh.	Below 88	From 93 to 101(*)	....
...	221	" " —100–150 mesh.	Below 58	At 61	At 76
(C)	221	Fine silica sand	Below 41	→	At 42
(D)	221	Silica dust	Below 56	→	At 58
(E)	221	Hydrated lime	Below 41	→	At 42
(F)	221	Talc (Georgia)	Below 76	From 81 to 101(*)	....
Produced from Mid-Continental Petroleum:					
(G)	225	None	Below 101 (*)		
Produced from Texas Petroleum:					
(H)	219	None	Below 30	At 37	At 42
(I)	225.5	Alberene flour (200–300 mesh.)	Below 30	From 37 to 50(*)	....
(J)	223.7	Dolomite flour	Below 30	At 37	At 46
Produced from Colombian Petroleum:					
...	225	None	Below 39	→	At 40–42
(K)	225	Slate flour	Below 84(*)	.....	....
...	225	Limestone flour	Below 64	From 71 to 84(*)	....
(L)	225	Dolomite flour	Below 54	From 59 to 84(*)	....
(M)	225	Hydrated lime	Below 30	At 39	At 43

(*) Test discontinued.
(†) Observation not conclusive.

TABLE CXIX

Compositions, Physical Properties, and Durabilities of Asphalt-Filler Mixtures

Specimen	Asphalt-Filler Mixture, per cent: Hard Asphalt	Asphalt-Filler Mixture, per cent: Soft Asphalt	Asphalt-Filler Mixture, per cent: Filler	Percentage of Hard Asphalt in Total Asphalt	Kind of Filler	Penetrations in 0.01 cm. units [a]: At 32 F., 200 g., 60 sec.	Penetrations in 0.01 cm. units [a]: At 77 F., 100 g., 5 sec.	Penetrations in 0.01 cm. units [a]: At 115 F., 50 g., 5 sec.	Durability: 1½ yr. Outdoor Exposure, Group Classification [b]	Durability: Life in Accelerated Test cycles
No. 1	47	18	35	72	Green slate flour, filler No. 1	6	9	13	No. I	62
No. 2	62	13	25	83		6	9	16	No. II	45
No. 3	73	12	15	86		7	9	17	No. II	37
No. 4	47	18	35	72	Dolomite, filler No. 2	6	10	18	No. II	37
No. 5	60	15	25	80		6	10	17	No. II	28
No. 6	73	12	15	86		7	10	17	No. III	28
No. 7	47	18	35	72	Dolomite, filler No. 3	6	9	15	No. II	40
No. 8	60	15	25	80		8	11	18	No. II	3
No. 9	73	12	15	86		7	10	17	No. III	28
No. 10	47	18	35	72	Limestone, filler No. 4	6	9	16	No. II	37
No. 11	60	15	25	80		6	10	17	No. II	43
No. 12	73	12	15	86		6	9	18	No. III	33
No. 13	47	18	35	72	Peach-bottom slate, filler No. 5	6	8	15	No. I	65
No. 14	60	15	25	80		7	10	16	No. I	62
No. 15	73	12	15	86		7	8	18	No. II	43
No. 16	47	18	35	72	Silica sand, filler No. 6	8	12	18	No. II	28
No. 17	68	7	25	90		6	11	15	No. II	28
No. 18	80	5	15	94		8	11	14	No. III	28
No. 19	47	18	35	72	Greenstone, filler No. 7	7	11	14	No. I	65
No. 20	60	15	25	80		7	10	16	No. II	46
No. 21	73	12	15	86		7	10	17	No. II	40
No. 22	—	65	35	0	Hydrated lime, filler No. 8	6	8	14	No. III	28
No. 23	—	75	25	0		8	11	21	No. III	28
No. 24	37	48	15	56		7	11	21	No. III	28
No. 25	36	29	35	55	Trap rock, filler No. 9	6	9	15	No. II	37
No. 26	53	22	25	70		7	9	17	No. II	43
No. 27	72	13	15	85		7	9	16	No. III	28
No. 28	13	72	15	15	Supercel, filler No. 10	8	10	22	No. II	40
No. 29	41	44	15	48	Mica, filler No. 11	7	10	17	No. I	65 +
No. 30	23	42	35	36	Foliated talc, filler No. 12	6	10	16	No. I	65
No. 31	44	31	25	58		7	9	18	No. I	65
No. 32	66	19	15	78		7	9	18	No. II	48
No. 33	49	16	35	75	Silica dust, filler No. 13	7	8	16	No. II	35
No. 34	62	13	25	83		7	8	16	No. II	33
No. 35	78	7	15	92		7	9	17	No. III	28
No. 36	40	25	35	62	Silica dust, filler No. 14	7	9	18	No. II	35
No. 37	54	21	25	72		6	9	16	No. II	30
No. 38	72	13	15	85		6	9	16	No. III	28
No. 39	49	16	35	75	Silica dust, filler No. 15	7	9	17	No. II	35
No. 40	64	11	25	85		7	10	17	No. II	35
No. 41	77	8	15	91		7	9	17	No. III	28
No. 42	33	32	35	51	Slate flour, filler No. 16	7	10	17	No. I	65
No. 43	50	25	25	67		7	10	17	No. II	51
No. 44	68	17	15	80		7	10	17	No. II	43
No. 45	44	21	35	68	Slate flour, filler No. 17	7	10	14	No. I	65
No. 46	58	17	25	78		7	10	15	No. I	65
No. 47	75	10	15	88		7	10	18	No. II	43
No. 48	43	22	35	66	Limestone, filler No. 18	7	10	18	No. II	45
No. 49	56	19	25	75		7	10	18	No. III	33
No. 50	75	10	15	88		7	10	19	No. III	28
No. 51	49	16	35	75	Limestone, filler No. 19	7	10	18	No. II	58
No. 52	64	11	25	85		8	10	17	No. II	48
No. 53	81	4	15	95		6	10	19	No. III	28
No. 54	100	—	—	100	.....	7	10	17	No. III	28
No. 55	75	25	—	75	.....	9	11	17	No. III	28
No. 56	50	50	—	50	.....	8	12	21	No. III	28
No. 57	25	75	—	25	.....	9	15	27	No. III	28

[a] The results reported are the average of 3 or 4 determinations.

[b] Group No. I.—Outdoor panels showing no cracks to the eye or with magnifying glass, or, at most, some of the panels of a set showing faint cracks to the eye or with a magnifying glass. Group No. II.—Outdoor panels showing cracks to the eye. Group No. III.—Outdoor panels showing wide cracks to the eye.

as they did in the accelerated cycle, and it can be reasonably assumed that 30 cycles in the accelerated test are equivalent to about 1½ years' outdoor exposure. The life of the coatings in the accelerated test are given in cycles in Table CXXI.

TABLE CXX

PHYSICAL PROPERTIES OF THE FILLERS

Filler	Kind of Filler	Particle Size of Filler			Oil-Absorption Ratio	Specific Gravity	Compacting Weight	Fineness Factor *f*
		Passing No. 200 Sieve, per cent	Passing No. 100 but Retained on No. 200 Sieve, per cent	Retained on No. 100 Sieve, per cent				
No. 1	Slate flour—Air floated No. 000 grade	81	17	1	0.7	2.86	1.35	1.1
No. 2	Dolomite	92	7	1	0.5	2.85	1.63	0.8
No. 3	Dolomite	85	14	1	0.4	2.88	1.73	0.7
No. 4	Limestone	91	8	1	0.4	2.76	1.62	0.7
No. 5	Peach-bottom slate—No 000 grade	79	16	5	0.8	2.98	1.40	1.1
No. 6	Silica sand	2	13	85	0.2[a]	2.69	1.74	0.6
No. 7	Greenstone	65	24	12	0.6	3.08	1.66	0.9
No. 8	Hydrated lime	99	2	..	1.3	2.39	0.83	1.9
No. 9	Trap rock	97	3	..	0.7	2.94	1.52	0.9
No. 10	Celite (Hy Flo Supercel)	100	.	.	2.9	2.52	0.30	7.4
No. 11	Mica	42	28	30	2.2[b]	3.20	0.51	5.3
No. 12	Foliated talc	100	trace retained on No. 200 sieve	.	1.1[b]	2.97	1.05	1.8
No. 13	Silica dust	67	26	7	0.5[b]	2.69	1.68	0.6
No. 14	Silica dust (prepared from filler No. 13)	100	.....	.....	0.5	2.69	1.63	0.7
No. 15	Silica dust (prepared from filler No. 13)	.....	100	.....	0.4	2.69	[c]	.....
No. 16	Slate flour (prepared from filler No. 5)	100	.....	.....	0.9	2.98	1.30	1.3
No. 17	Slate flour (prepared from filler No. 5)	.....	100	.....	0.7	2.98	[c]	.....
No. 18	Limestone (prepared from filler No. 4)	100	.....	.....	0.5	2.76	1.63	0.7
No. 19	Limestone (prepared from filler No. 4)	.....	100	.....	0.5	2.76	[c]	.....

[a] Does not possess plastic properties, but merely wets.
[b] Not a sharp end-point.
[c] Insufficient material for test.

TABLE CXXI

ORDER OF DURABILITY OF ASPHALT-FILLER MIXTURES

Specimen	Composition	Durability, number of cycles
	GROUP No. I	
No. 29	15 per cent Mica Filler No. 11	65+
No. 45	35 per cent Slate Filler No. 17	65
No. 30	35 per cent Talc Filler No. 12	65
No. 31	25 per cent Talc Filler No. 12	65
No. 13	35 per cent Slate Filler No. 5	65
No. 42	35 per cent Slate Filler No. 16	65
No. 46	25 per cent Slate Filler No. 17	65
No. 1	35 per cent Slate Filler No. 1	62
No. 19	35 per cent Greenstone Filler No. 7	65
No. 14	25 per cent Slate Filler No. 5	62
	GROUP No. III	
No. 6	15 per cent Dolomite Filler No. 2	28
No. 9	15 per cent Dolomite Filler No. 3	28
No. 12	15 per cent Limestone Filler No. 4	33
No. 18	15 per cent Sand Filler No. 6	28
No. 27	15 per cent Trap rock Filler No. 9	28
No. 35	15 per cent Silica Filler No. 13	28
No. 38	15 per cent Silica Filler No. 14	28
No. 41	15 per cent Silica Filler No. 15	28
No. 49	25 per cent Limestone Filler No. 18	33
No. 53	15 per cent Limestone Filler No. 19	28
No. 54	Asphalt softening point 108° C.	28
No. 55	Asphalt softening point 104° C.	28
No. 56	Asphalt softening point 99.5° C.	28
No. 57	Asphalt softening point 94° C.	28
No. 50	15 per cent Limestone Filler No. 18	28
No. 22	35 per cent Hydrated lime Filler No. 8	28
No. 23	25 per cent Hydrated lime Filler No. 8	28
No. 24	15 per cent Hydrated lime Filler No. 8	28
	GROUP No. III	
No. 25	35 per cent Trap rock Filler No. 9	43
No. 20	25 per cent Greenstone Filler No. 7	46
No. 32	15 per cent Talc Filler No. 12	48
No. 43	25 per cent Slate Filler No. 16	51
No. 4	35 per cent Dolomite Filler No. 2	37
No. 7	35 per cent Dolomite Filler No. 3	40
No. 10	35 per cent Limestone Filler No. 4	43
No. 2	25 per cent Slate Filler No. 1	45
No. 15	15 per cent Slate Filler No. 5	43
No. 28	15 per cent Supercel Filler No. 10	40
No. 33	35 per cent Silica Filler No. 13	35
No. 36	35 per cent Silica Filler No. 14	35
No. 39	35 per cent Silica Filler No. 15	35
No. 40	25 per cent Silica Filler No. 15	35
No. 47	15 per cent Slate Filler No. 17	43
No. 51	35 per cent Limestone Filler No. 19	58
No. 52	25 per cent Limestone Filler No. 19	48
No. 5	25 per cent Dolomite Filler No. 2	28
No. 8	25 per cent Dolomite Filler No. 3	37
No. 11	25 per cent Limestone Filler No. 4	37
No. 26	25 per cent Trap rock Filler No. 9	37
No. 3	15 per cent Slate Filler No. 1	37
No. 21	15 per cent Greenstone Filler No. 7	40
No. 34	25 per cent Silica Filler No. 13	33
No. 37	25 per cent Silica Filler No. 14	30
No. 44	15 per cent Slate Filler No. 16	43
No. 48	35 per cent Limestone Filler No. 18	45
No. 16	35 per cent Sand Filler No. 6	28
No. 17	25 per cent Sand Filler No. 6	28

Test 103. Modified Accelerated Weathering Test. The Bureau of Standards, Washington, D. C., has recommended a modification which consists in exposing a series of panels aggregating about 180 sq. in. of surface to the carbon arc for 18 hours, followed by immersion of the panels in distilled water at room temperature for 5 to 6 hours. After periods of 26 cycles, the water solution containing the soluble decomposition products is evaporated to dryness and the residue weighed. The following results have been reported:

TABLE CXXII

BEHAVIOR OF PETROLEUM-ASPHALTS IN STANDARD AND MODIFIED ACCELERATED TESTS

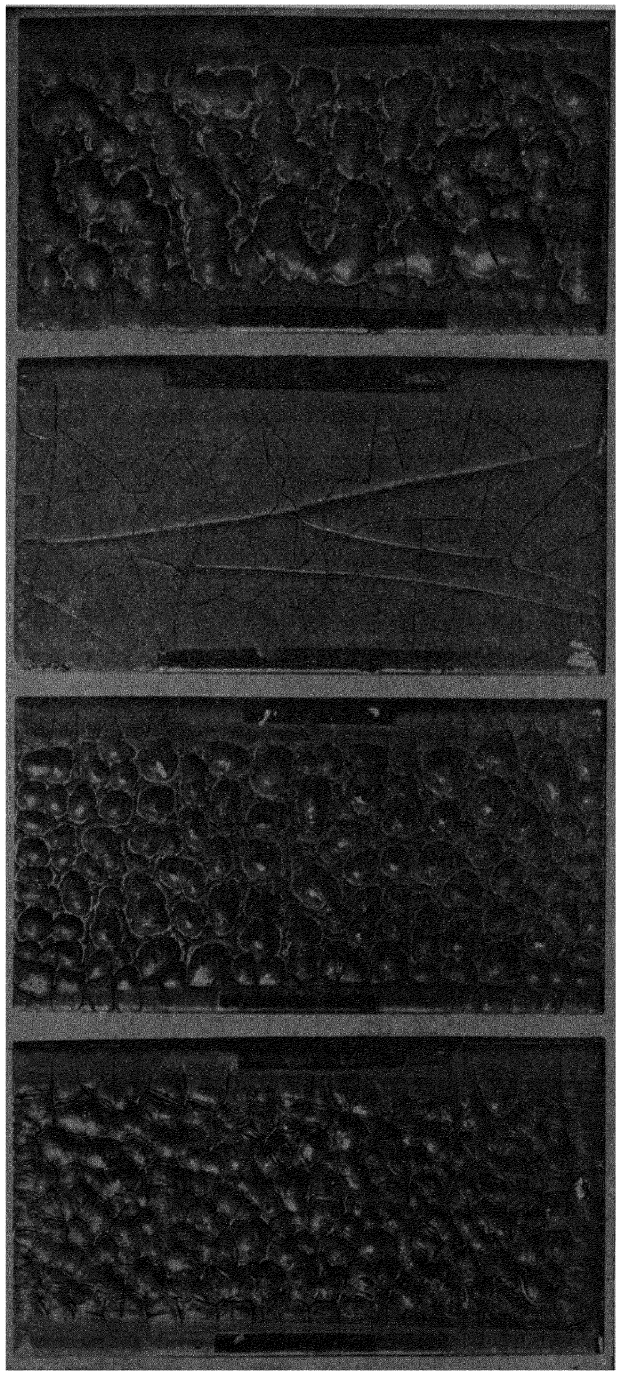
Blown Asphalt from:	Mid-Continental Petroleum	Mexican Petroleum	Venezuelan Petroleum	Californian Petroleum
Standard Accelerated Test:				
Life in cycles	56	26	34	29
Appearance at end of test	Smooth	Pronounced hummocks	Hummocks	Pronounced hummocks
Modified Accelerated Test (a):				
Life in cycles	179 (b)	87	47	57
Appearance at end of test	Smooth; some grooving	Smooth	Smooth; slight grooving	Smooth; some grooving
Water-soluble Products (grams per 100 sq. in. surface):				
1st—26 cycles (Total 26 cycles)	1.161	1.317	1.350	1.583
2nd—26 cycles (Total 52 cycles)	1.311	1.311	1.606	1.694
..... 9 cycles (Total 61 cycles)	0.500	0.444	0.400	0.655
3rd—26 cycles (Total 87 cycles)	1.233	1.322	1.667	2.411
4th—26 cycles (Total 113 cycles)	0.778	0.889	1.167	1.722
5th—26 cycles (Total 139 cycles)	0.667	0.722	1.000	1.222
6th—26 cycles (Total 165 cycles)	0.556	0.556	0.778	1.167
7th—26 cycles (Total 191 cycles)	0.500(c)	.....	.....	.....
Total in 165 cycles	6.206	6.561	7.968	10.454
Appearance at end of 191 cycles	Asphalt layer intact	Slight loss of asphalt layer	Slight loss of asphalt layer	About third of asphalt layer lost

(a) 12 panels of each specimen subjected to Modified Accelerated Test, exposing about 180 sq. in. surface of asphalt, weight about 140 grams.

(b) Started to crack at 125 cycles.

(c) Not included in total of 6.206.

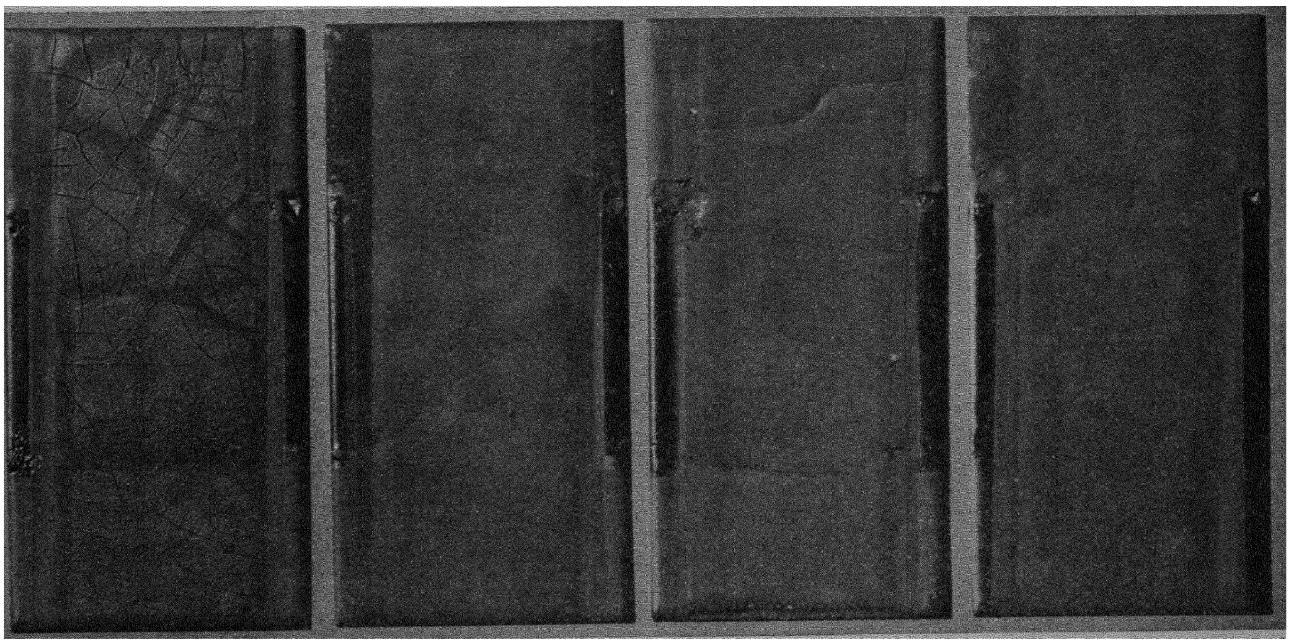
Fig. 331 shows the appearance of the panels at the end of the standard accelerated test, Fig. 332 shows the appearance of the specimens at the end of 165 modified accelerated cycles, and Fig. 333 at the end of 191 modified accelerated cycles.



Courtesy Bureau of Standards

Venezuelan Mexican Mid-Continental Californian

FIG. 331.—Standard Accelerate Weathering Cycles. Appearance at end of test.



Courtesy Bureau of Standards

Venezuelan Mexican Mid-Continental Californian

FIG. 332.—Modified Weathering Cycles. Appearance at end of test.

Courtesy Bureau of Standards

Californian

Mid-Continental

Mexican

Venezuelan

FIG. 333.—Exposed for 191 Modified Cycles.

TEMPERATURE CONVERSION TABLE

Fahrenheit to Centigrade

F.°	0	10	20	30	40	50	60	70	80	90	Fractional Parts	
	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	F.°	C.°
0	−17.7	−12.2	−6.6	−1.1	+4.4	+10.0	+15.5	+21.1	+26.6	+32.2	1	0.5
100	37.7	43.3	48.8	54.4	60.0	65.5	71.1	76.6	82.2	87.7	2	1.1
200	93.3	98.8	104.4	110.0	115.5	121.1	126.6	132.2	137.7	143.3	3	1.6
300	148.8	154.4	160.0	165.5	171.1	176.6	182.2	187.7	193.3	198.8	4	2.2
400	204.4	210.0	215.5	221.1	226.6	232.2	237.7	243.3	248.8	254.4	5	2.7
500	260.0	265.5	271.1	276.6	282.2	287.7	293.3	298.8	304.4	310.0	6	3.3
600	315.5	321.1	326.6	332.2	337.7	343.3	348.8	354.4	360.0	365.5	7	3.8
700	371.1	376.6	382.2	387.7	393.3	398.8	404.4	410.0	415.5	421.1	8	4.4
800	426.6	432.2	437.7	443.3	448.8	454.4	460.0	465.5	471.1	476.6	9	5.0
900	482.2	487.7	493.3	498.8	504.4	510.0	515.5	521.1	526.6	532.2		
1000	537.7	543.3	548.8	554.4	560.0	565.5	571.1	576.6	582.2	587.7		

Centigrade to Fahrenheit

C.°	0	10	20	30	40	50	60	70	80	90	Fractional Parts	
	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°		
−0	+32	+14	−4	−22	−40	−58	−76	−94	−112	−130	C.°	F.°
+0	32	50	68	86	104	122	140	158	176	194	1	1.8
100	212	230	248	266	284	302	320	338	356	374	2	3.6
200	392	410	428	446	464	482	500	518	536	554	3	5.4
300	572	590	608	626	644	662	680	698	716	734	4	7.2
400	752	770	788	806	824	842	860	878	896	914	5	9.0
500	932	950	968	986	1004	1032	1040	1058	1076	1094	6	10.8
600	1112	1130	1148	1166	1184	1202	1220	1238	1256	1274	7	12.6
700	1292	1310	1328	1346	1364	1382	1400	1418	1426	1454	8	14.4
800	1472	1490	1508	1526	1544	1562	1580	1598	1616	1634	9	16.2
900	1652	1670	1688	1706	1724	1742	1760	1778	1796	1814		
1000	1832	1850	1868	1886	1904	1922	1940	1958	1976	1994		

Black figures indicate recurring decimals

Examples: 567° F. = 293.33 + 3.988 = 297.22° C.; − 85° C. = − 112 − 9.0 = − 121° F.

BIBLIOGRAPHY

(A) GENERAL TREATISES

Abraham, Herbert; "Asphalts and Allied Substances"; 1st Edition 1918; 2nd Edition 1920; 3rd Edition 1929; 4th Edition 1937; D. Van Nostrand Co., Inc; New York.

Alberghina, Giacomo; "L'asfalto e il problema dei carburanti in Italia"; 1937; Antica libreria Reber; Palermo.

Alvarez, H. H.; "Combustibles sólidos de la República Argentine"; 1920; Talleres gráficos del Ministerio de agricultura de la nación; Buenos Aires.

Ascione, Ernesto; "L' industria dell asfalto" 1913; Dr. Francesco Vallardi Publishing Co.; Milan.

Attwool, A. W.; "Trinidad Lake Asphalt"; 1930; The Limmer & Trinidad Lake Asphalt Co., Ltd.; London.

Attwool, A. W., and Broome, D. C.; "Trinidad Lake Asphalt"; 1935; The Bayard Press; London.

Beckwith, Arthur; "Report on Asphalt and Bitumen"; 1868; U. S. Government Printing Office; Washington, D. C.

Boorman, T. H.; "Asphalts—Their Sources and Utilizations"; 1908; Wm. T. Comstock; New York.

Bösenberg, H.; "Die Praxis des neuzeitlichen Asphaltstrassenbaus", Vol. I, "Untersuchung und Auswahl des Bitumens"; 1928; Brend'amour, Simhart & Co.; Munich.

Breynaert, E. F.; "Le gisement asphaltique du Val de Travers"; 1912; H. Dunod et H. Pinat; Paris.

Britton, H.; "Bibliography of Petroleum and Allied Substances" (Bulletin No. 290); 1922-1923; U. S. Geological Survey, Government Printing Office; Washington, D. C.

Broome, D. C.; "The Testing of Bituminous Mixtures" (Vol. II of Roadmakers' Library); 1934; Edward Arnold & Co.; London.

Burroughs, E. H.; "Bibliography of Petroleum and Allied Substances"; Bulletin No. 149, 1915; Bulletin No. 165, 1916; Bulletin No. 180, 1917; Bulletin No. 189, 1918; Bulletin No. 216, 1919-1920; Bulletin No. 220, 1921; U. S. Geological Survey, Government Printing Office; Washington, D. C.

Cannon, Jr., J. Q., "Standards and Specifications for Nonmetallic Minerals and Their Products", Miscellaneous Publication No. 110; Apr. 1930; National Bureau of Standards, U. S. Department of Commerce; Government Printing Office; Washington, D. C.

Chabrier, Ernest; "The Applications of Asphalt"; 1876; William Clowes & Sons; London.

Chambrier, Paul de; "Historique de Péchelbronn 1498-1918"; Attinger Frères; Paris.

Colonial Geologists; "St. Domingo Petroleum and Trinidad Asphalt"; 1860; Longmans & Co.; London.

Cooper, A. S.; "The Genesis of Petroleum and Asphaltum in California" (Bulletin No. 16); 1899; California State Mining Bureau; Sacramento, Cal.

Cross, Roy; "Petroleum, Asphalt and Natural Gas"; Bulletins No. 14, 1918; No. 15, 1920; No. 16, 1922; No. 17, 1924; No. 25, 1928; No. 25, Revised, 1931; Kansas City Testing Laboratory; Kansas City, Mo.

Dammer, Bruno, and Tietze, Oskar; "Die nutzbaren Mineralien, mit ausnahme der Erze und Kohlen"; Vol. II, "Die natürlichen Bitumina", by *W. Wunstorf;* 2nd Edition 1928; Verlag von Ferdinand Enke; Stuttgart.

Danby, Arthur; "Natural Rock Asphalts and Bitumens"; 1913; D. Van Nostrand Co.; New York.

Daubrée, Auguste; "Mémoire sur le gisement de bitume, de lignite et de sel, dans le terrain tertiare de Bechelbronn et de Lobsann (Bas-Rhin)"; 1867; H. Dunod et Cie; Paris.

Davin, Étienne E.; "Le Mazout"; 1923; Revue les Matières Grasses, Pétrol et ses Dérivés; Paris.

Degermann, M. F.; "Une ancienne source de bitume en Alsace"; 1894; (Extrait du Bulletin de la Société d'histoire de Colmar 1891-1894); Impr. de Decker; Colmar.

Delano, W. H.; "Twenty Years' Practical Experiences of Natural Asphalt and Mineral Bitumen"; 1893; E. F. & N. Spon, Ltd.; London and New York.

Doelter (y Cisterich), C. A., and *Leitmeier, H.;* "Handbuch der Mineralchemie"; Section on "Asphalt" (Vols. III and IV) by *Albrecht von Skopnik;* 1931; Verlag von Theodor Steinkopff; Dresden.

Dücker, F. F. von; "Petroleum und Asphalt in Deutschland"; 1881; Verlag von J. C. C. Bruns; Minden.

Ekstrand, Charles; "Asphalt; its History, Manufacture and Uses"; 1915; Warner-Quinlan Asphalt Co.; New York.

Eldridge, G. H.; "The Uintaite (Gilsonite) Deposits of Utah"; Vol. IV, Part I of the 17th Annual Report of the U. S. Geological Survey; 1896; U. S. Government Printing Office; Washington, D. C.

Eldridge, G. H.; "The Asphalt and Bituminous Rock Deposits of the United States"; Part I of the 22nd Annual Report of the U. S. Geological Survey; 1901; U. S. Government Printing Office; Washington, D. C.

Ells, S. C.; "Preliminary Report on the Bituminous Sands of Northern Alberta"; Bulletin No. 281; 1914; Canadian Government Printing Bureau; Ottawa.

Engineering Standards' Committee; "British Standard Nomenclature of Tars, Pitches, Bitumens and Asphalts, and British Standard Specifications for Tar and Pitch"; 1916; Crosby, Lockwood & Son; London.

d' Erynys, Eirinis; "Dissertation sur l'asphalte ou ciment naturel, découvert depuis quelques années au Val de Travers"; 1721; P.–N. Lottin; Paris.

Fischer, Emil J.; "Die natürlichen und künstlichen Asphalte und Peche"; Vol. XIX of "Technischen Fortschrittsberichte"; 1928; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Fischer, Emil J.; "Anwendung organischer Lösungsmittel bei der Analyse organisch-technischer Rohstoffe und Rohstoffgemische"; 1932; Verlag von Wilhelm Knapp; Halle (Saale).

Fischer, Emil J.; "Untersuchung von Asphalt- und Pechgemengen"; 1932; Verlag von Wilhelm Knapp; Halle (Saale).

Fischer, Emil J.; "Industrieteere und verwandte Produkte"; 1933; Verlag von Wilhelm Knapp; Halle (Saale).

Forbes, R. J.; "The Story of Bitumen" (From the Earliest Times up to the Nineteenth Century A. D.); 1934; Shell Oil Co. (N. V. de Bataafsche Petroleum Maatschappij); Amsterdam, Holland.

Forbes, R. J.; "Bitumen and Petroleum in Antiquity"; 1936; E. J. Brill; Leiden, Holland.

Fournel, Henri; "Notice sur la pierre asphaltique du Val-de-Travers dans le Canton de Neufchâtel, avec une préface de M. le Comte de Sassenay"; 1838; Ladrange; Paris.

Friese, Wilhelm; "Die Asphalt- und Teerindustrie"; 1908; Dr. Max Jänecke Verlagsbuchhandlung; Hannover.

Gaetz, H. T.; "Water-white Hydrocarbons from Trinidad Asphalt"; 1936; Supplemental Volume "General Discussion"; 1936; Grafton Publishing Co.; Los Angeles, Cal.

Gesner, Abraham; "A Practical Treatise on Coal, Petroleum, and Other Distilled Oils"; 1st Edition, 1861; 2nd Edition, 1865; Baillière Brothers; New York.

Giardina, Pietro; "Le miniera di asfalto"; Revised Edition 1923; Union Tipografico-Editrice Torinese; Turin, Italy.

Gounot, A.; "Notes sur les mines de bitume exploitées en Albanie"; 1903; H. Dunod et Cie.; Paris.

Halleck, H. Wagner; "Bitumen, Its Varieties, Properties and Uses"; 1841; Peter Force; Washington, D. C.

Hauptausschutz für Asphalt- und Teerforschung; "DIN–1995; Teil I— Vorschriften für die Prüfung von Asphalt und Teer (als reine Bindemittel); Teil II—Vorschriften für die Prüfung von Asphalt und Teer enthaltenden Massen"; 1929; "DIN–1996; Vorschriften für die Lieferung von Asphalt und Teer, sowie von Asphalt und Teer enthaltenden Massen"; 1929; Vereinigung der technischen Oberbeamten Deutscher Städte Kassel; Beuth-Verlag; Berlin.

Hauptausschutz für Asphalt- und Teerforschung "DIN–1995 and DIN–1996; Neubearbeitete Teilvorschriften"; Heft 1, Jan. 1931; Heft 2, Jan. 1931; Heft 3, Jan. 1931; Heft 4, Jan. 1931; Heft 5, Jul. 1932; Heft 6, Jul. 1932; Heft 7, Jan. 1932; also Sachregister; Beuth-Verlag; Berlin.

Henning, D. H.; "Mineral-asphaltstein der Mine zur Limmer, Feldmark Velber bei Hannover; nebst eine kurzen Anleitung zur Anwendung desselben bei verschiedenen Bauten"; 1866; Verfasser; Limmer bei Hannover.

Héricart de Thury, L. E. F.; "Notice sur les mines d'asphalte, bitume, et lignites de Lobsann, Arrondissement de Weissembourg (Départment du [illegible] Rhin)"; 1838; F. Didot Frères; Paris.

Hirz, Heinrich, and *Pothmann, Wilhelm;* "Jahrbuch der deutschen Braunkohlen-, Steinkohlen-, Kali- und Erzindustrie, der Salinen, des Erdöl, und Asphaltbergbaus, 1929"; 20th Year, 1929; Verlag von Wilhelm Knapp; Halle (Saale).

Hoeffel, Johannes Theophilius; "Historia balsami mineralis, Alsatici seu petrolei, vallis sancti Lamperti—Der Hanauische Erd-Balsam, Lampertslocher Oel- oder Baechel-Brunn"; 1734; Les Successeurs de Johannis Pastorius; Strasbourg.

Hoofdcommissie voor de Normalisatie in Nederland; "Keuringsvoorschriften voor Bitumineuze Bouwstoffen (K.V.B.B. 1930)"; N–1013; Effective Apr. 1930; Hoofdcommissie voor de Normalisatie in Nederland; Centraal Normalisatie Bureau; 's-Gravenhage, Holland.

Huguenet, Isidore; "Considerations générales sur l'origine et la formation des asphaltes"; 1847; L. Mathias; Paris.

Huguenet, Isidore; "Asphaltes et Naphtes; Considérations générales sur l'origine et la formation des bitumes fossiles, et leur emploi"; 1st Edition, 1847; 2nd Edition, 1852; Librairie Scientifiques-Industrielle, Paris; 2nd Edition, Revised, 1853; Verlag von Bernhard Friedrich Voigt; Leipzig.

Huguenet, Isidore; "Ueber den Asphalt" (Translated from the French); 1853; Verlag von Bernhard Friedrich Voigt; Leipzig.

Hutchinson, L. L.; "Asphalt and Petroleum in Oklahoma" (Bulletin No. 2); 1911; Oklahoma Geological Survey; Leader Printing Co.; Guthrie, Okla.

Jaccard, Auguste; "Le pétrole, l'asphalte et le bitume"; 1895; Germer Baillière & Cie.; Paris.

Jackson, C. T.; "Book of Trinidad"; 1904; Muir, Marshall & Co.; Trinidad.

Jillson, W. R.; "Economic Papers on Kentucky Geology" (Series 6, Vol. II); 1921; The Kentucky Geological Survey; Frankfort, Ky.

J. V. Z.; "Asphalt, its Preparation and Application"; 1875; Private Press; Boston, Mass.

Kayser, R.; "Untersuchung über natürliche Asphalte"; 1879; Fr. Korn Verlag; Nürnberg.

Köhler, Hippolyt, "Die Chemie und Technologie der natürlichen und künstlichen Asphalte"; 1st Edition, 1904; *Köhler, Hippolyt,* and *Graefe, Edmund;* 2nd Edition, 1913; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Kovács, Jenö; "Ueber Asphalt, sein Vorkommen, seine Verwendung und einschlägige Untersuchungen"; 1901; Pester Buchdruckerei A.–G.; Budapest.

Křepelka, V.; "Ueber Methoden der Untersuchung der natürlichen von den künstlichen Asphalte"; 1904; Inaugural-Dessertation; Zürich.

Langton, H. M.; "Blacks and Pitches"; 1925; D. Van Nostrand Co.; New York.

Le Bel, J.-A.; "Notice sur les gisements du pétrole à Péchelbronn"; 1885; Veuve C. Decker; Colmar.

Lévy–Alexandre, G.; "Bitumes et asphaltes; leurs principales applications dans les travaux"; 1920; L. Eyrolles; Paris.

Lindenberg, Felix; "Die Asphalt-Industrie"; 1908; A. Hartleben's Verlag; Vienna and Leipzig.

Lynch, W. F.; "Dead Sea Asphalt"; 1852; Official Report of the U. S. Expedition to Explore the Dead Sea and the River Jordan; U. S. Government Printing Office; Washington, D. C.

Malenkovic, Basilius; "Die Asphaltfrage, inbesondere die Nomenklaturfrage, vom Standpunkte des Hochbau- und Strassenbau-Ingenieurs"; 1906; Stähle & Friedel; Stuttgart.

Mallison, Heinrich; "Teer und Pech"; 1924; Verlag von Wilhelm Knapp; Halle (Saale).

Mallison, Heinrich; "Teer, Pech, Bitumen und Asphalt"; 1926; Verlag von Wilhelm Knapp; Halle (Saale).

Malo, Edouard; "L'asphalt, ce qu'il est, d'ou il vient, comment on l'emploie"; 1905; A. Rey; Lyon, France.

Malo, Léon; "Note sur l'asphalte, son origine, sa préparation, ses applications, suivie de divers documents sur la matière"; 1863; Baudry et Cie.; Paris.

Malo, Léon; "L'asphalte, son origine, sa préparation, ses applications"; 1st Edition 1866; 2nd Edition 1888; 3rd Edition 1898; Baudry et Cie.; Paris.

Malo, Léon; "Ueber Asphalt" (Translated from the French); 1882; F. Woas; Paris.

Malo, Léon; "Treatise on the Use of French Rock Asphalt (Seyssel)"; 1884; E. H. Wootton; New York.

Marcusson, Julius, "Die natürlichen und künstlichen Asphalte"; 1st Edition, 1921; *Marcusson, Julius; Burchartz, H.;* and *Wilke, P.;* 2nd Edition, 1931; Verlag von Wilhelm Engelmann, Leipzig.

Martin, Oskar; "Untersuchungen über die Bindefähigkeit bituminöser Stoffe und deren Messung"; 1936; Allgemeiner Industrie-Verlag G.m.b.H.; Berlin.

Mazzocchi, Luigi; "Asfalto, bitume e catrame"; 1920; Ulrico Hoepli; Milan.

Metzger, Hans; "Starrpunkt und Viscosität bituminöser Stoffe"; 1930; Verlag von Wilhelm Knapp; Halle (Saale).

Narcy, Phillippe; "Les bitumens"; 1898; P. Vicq-Dunod et Cie.; Paris.

Nellensteyn, F. J.; "Bereiding en Constitutie van Asphalt" (Dissertation Delft); 1923; J. Deterre & Soon; Aachen, Holland.

Nöthling, Ernst (Wilhelm Jeep); "Der Asphalt und seine Anwendung in der Technik"; 1st Edition 1867; 2nd Edition 1899; Verlag von Bernhard Friedrich Voigt; Weimar.

Oberbach, J. H., and *Pauer, O.;* "Ueber die Zusammensetzung von Erdölasphalten"; 1937; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Orton, Edward; "Occurrence of Petroleum, Natural Gas and Asphalt Rock in Western Kentucky"; 1888-1889; Geological Survey of Kentucky; Louisville, Ky.

Payen, Anselme; "Mémoire sur les bitumes, leur exploitation et leur emploi, à l'occasion d'un rapport sur les produits bitmineux des mines de Lobsann"; 1824; Thomine; Paris.

Peckham, S. F.; "Solid Bitumens"; 1909; Myron C. Clark Publishing Co.; New York and Chicago.

Pennybacker, J. E.; "Asphalt – Nature's Most Versatile Product"; 1936; The Asphalt Institute; New York.

Phillips, W. B.; "Coal, Lignite and Asphalt Rocks" (Bulletin No. 3); 1902; University of Texas Mineral Survey; Austin, Texas.

Pompucci, Alessandro; "Le analisi dei bitumi"; 1937; La poligrafica; Pesaro.

Posewitz, Theodor; "Petroleum und Asphalt in Ungarn"; 1907; Buchdruckerei des Franklin-Vereins; Budapest.

Rebstein, Otto; "Beiträge zur Kenntnis der chemischen Zusammensetzung Schweizerischer Bitumina"; 1928; Allgemeiner Industrie-Verlag G.m.b.H.; Berlin.

Richardson, Clifford; "On the Nature and Origin of Asphalt" (Bulletin No. 2); 1898; Barber Asphalt Paving Co.; Philadelphia.

Robinson, E.; "Dead Sea Asphalt"; 1841; Biblical Researches; London.

Seyer, W. F.; "A Chemical Investigation of the Asphalt in the Sands of Northern Alberta" (Publication No. 32, Series 3); 1922; McGill University; Montreal.

Späte, F.; "Die Bituminierung"; 1907; Dissertation; Berlin.

Spielmann, Jacob Reinhold; "Mémoire sur le bitume d'Alsace"; 1758; Reproduit: Collection académique, partie étrangère, Tome 9, pp. 405, 410 et 415; Académie de Berlin, Vol. XIV; 1758; Berlin.

Spielmann, P. E.; "Bituminous Substances"; 1925; Ernest Benn, Ltd.; London.

Sporny, Josef; "Der Asphalt und die Bitumina"; 2nd Edition 1878; St. Petersburg.

Standardization of Tar Products Tests Committee; "Standard Methods for Testing Tar and Its Products"; 1929; W. Heffer & Sons, Ltd.; Cambridge, England.

Swoboda, Julius; "Der Asphalt und seine Verwendung"; 1904; Verlag von Leopold Voss; Hamburg and Leipzig.

Tausz, Jenö, and *Lüttgen, A.;* "Schnell-methode zur Bestimmung von Hartasphalt"; 1919; Verlag für Fachliteratur G.m.b.H. (A. Paul & Co.); Berlin.

Thöenes, Dirk; "Het Ontstaan van Asfalt-Bitumen"; 1936; Drukkerij Waltman; Delft, Holland.

Volck, J.; "Hanauischen Erdbalsams oder weichen Agsteins Beschreibung, welcher bei Lampertsloch gefunden wird"; 1st Edition 1615; 2nd Edition 1625; Strassburg.

Warren, G. C., "The Part of the Warrens in the Development of Coal Tar, Petroleum Oil and Asphalt"; 1928; Warren Bros. Co.; Philadelphia.

Wauters, J.; "Los esquistos bituminosos de Cerro Largo y los ensayos practicados en el laboratorio del instituto en 1917"; 1924; Imp. del Comercio Calle Piedras; Montevidio, Chile.

Weil, S. F.; "Ueber Asphalt und verwandte Industrien"; 1910; Verlag von Eisenstein; Vienna and Leipzig.

Whitmore, W. H.; "Results of Destructive Distillation of Bituminous Substances"; 1860; Henry W. Dutton & Sons; Boston, Mass.

Woas, Franz; "Der Asphalt, seine Geschichte, Gewinnung und Verwendung"; 1880; Polytechnische Buchandlung (A. Seidel); Berlin.

Wurtz, Henry; "Report on a Mineral Formation in West Virginia"; 1865; Francis & Loutrell; New York.

Zetter, J. T.; "Der Asphalt und seine Verwendung in der Bautechnik"; 1880; Druck von Orell Füssli; Zürich.

(B) MINERAL WAXES

Berlinerblau, Josef; "Das Erdwachs, Ozokerit und Ceresin"; 1897; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Better, E. J., and *Davidsohn, J.;* "Taschenbuch für die Wachindustrie"; 1932; Wissenschaftliche Verlagsgesellschaft m.b.H.; Stuttgart.

Braun, E.; "Wachse und ihre Verwendung"; 1926; Verlag Max Jänecke; Leipzig.

Burgmann, Arthur; "Petroleum und Erdwachs"; (Vol. 72 of Chemisch-technische Bibliothek); 2nd Edition 1897; A. Hartleben's Verlag; Vienna and Leipzig.

Fischer, E. J.; "Wachse, wachsähnliche Stoffe und technische Wachsgemenge" (Technische Fortschrittsberichte Vol. XXXI, edited by *B. Rassow*); 1934; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Gregorius, Rudolf; "Erdwachs (Zeresin), Paraffin und Montanwachs, deren Darstellung und Verwendung" (Vol. 303 of Chemisch-technische Bibliothek); 1908; A. Hartleben's Verlag; Vienna and Leipzig.

Gregorius, Rudolf; "Mineral Waxes, Their Preparation and Uses" (Translated from the German by Chas. Salter); 1908; Scott, Greenwood & Son; London.

Grosse, W.; Die volkwirtschaftliche Bedeutung der sächsische-thüringischen Braunkohlenteer- und Montanwachsindustrie"; 1923; Verlag von Wilhelm Knapp; Halle (Saale).

Ivanovszky, Leo; "Ozokerit und verwandte Stoffe"; Vol. I: "Einführung in die Rohstofflehre und Technologie der wachsverarbeitenden Industrie, unter bes. Berücks. des Ozokerits"; Vol. II: Part 1, "Die Mineralwachse, usw."; Part 2, "Synthetische Wachse, usw." (Chemisch-technische Bibliothek, Vols. 397, 398 and 401); 1935; A. Hartleben's Verlag; Vienna and Leipzig.

Lach, Béla; "Die Zeresinfabrikation" (Vol. 22 of Monographien über chemisch-technische Fabrikationsmethoden); 1911; Verlag von Wilhelm Knapp; Halle (Saale).

Lüdecke, E.; "Die Wachse und Wachskörper" (Vol. VII of Monographien aus dem Gebiete der Fett-Chemie); 1926; Wissenschaftliche Verlagsgesellschaft m.b.H.; Stuttgart.

Muck, Josef; "Der Erdwachsbergbau in Boryslaw"; 1903; Verlag von Julius Springer; Berlin.

Sedna, Ludwig; "Das Wachs und seine technische Verwendung" (Vol. 132 of Chemisch-technische Bibliothek); 1st Edition 1886; 2nd Edition 1905; 3rd Edition 1919; A. Hartleben's Verlag; Vienna, Budapest and Leipzig.

(C) WOOD TARS AND PITCHES

Bergström, H. O. V., and *Van Wesslén, J. C.;* "Om Träkolning"; 1918; A.B.G. Tissell, Stockholm.

Brown, N. C.; "Forest Products: Their Manufacture and Use"; 1918; John Wiley & Sons, Inc., New York.

Bugge, Günther; "Industrie der Holzdestillationsprodukte" (Vol. XV of Technische Fortschrittsberichte); 1927; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Bunbury, H. M.; "The Destructive Distillation of Wood"; 1923; Benn Brothers, Ltd.; London.

Bunbury, H. M.; "Die trockene Destillation des Holzes" (Translated from the English by Wilhelm Elsner); 1925; Verlag von Julius Springer; Berlin.

Dumesny, Paul, and *Noyer, J.;* "L'industrie chimique des bois, leurs dérivés et extraits industriels"; 1st Edition (1 Vol.); 1906; B. Tignol, Paris; 2nd Edition (2 Vols.); 1925; Gauthier-Villars et Cie.; Paris.

Dumesny, Paul, and *Noyer, J.;* "Wood Products" (translated from the French); 1921; Scott, Greenwood & Son; London.

Gibbons, L. W.; "The Utilization of Hardwood Waste"; 1921; H. J. Baker & Bro.; New York.

Harper, W. B.; "Utilization of Wood Waste by Distillation"; 1908; D. Van Nostrand Co.; New York.

Harper, W. B.; "Die Destillation industrieller und forstwirtschaftlicher Holzabfälle" (Translated from the English); 1909; Verlag von Julius Springer; Berlin.

Hawley, L. F.; "Wood Distillation"; 1923; The Chemical Catalog Co. Inc.; New York.

Hawley, L. F.; "Holzdestillation" (Translated from the English by A. A. Schrieber); 1926; Verlag von Julius Springer; Berlin.

Hubbard, E.; "Die Verwertung der Holzabfälle" (Vol. 144 of Chem.-techn. Bibliothek); (4th Edition) 1921; A. Hartleben's Verlag; Vienna.

Klar, M.; "Technologie der Holzverkohlung"; 1st Edition, 1903; 2nd Edition, 1909; 3rd Edition, 1921; Verlag von Julius Springer; Berlin.

Klar, M.; "Technologie de la distillation du bois" (Translated from the German by Adolf Jouve); 1925; Ch. Béranger; Paris.

Klar, M.; "The Technology of Wood Distillation" (Translated from the German by Alexander Rule); 1927; D. Van Nostrand Co. Inc.; New York.

Kollmann, F.; "Technologie des Holzes"; 1936; Verlag von Julius Springer; Berlin.

Schweitzer, Victor; "The Distillation of Resins"; 1906; D. Van Nostrand Co. Inc.; New York.

Thenius, Georg; "Das Holz und seine Destillationsprodukte"; 1st Edition, 1909; 2nd Edition, 1912; 3rd Edition, 1922; A. Hartleben's Verlag; Vienna and Leipzig.

(D) PEAT, LIGNITE AND SHALE TARS AND PITCHES

Alderson, V. C.; "The Shale Oil Industry"; 1920; Fred. A. Stokes & Co.; New York.

Bertelsmann, W.; "Lehrbuch der Leuchtgasindustrie" (Vols. I and II); 1911; Verlag von Ferdinand Enke; Stuttgart.

Beyschlag, Rudolf; "Neuer und alte Wege der Braunkohlen- und Schieferverschwelung"; (2nd Edition) 1920; Verlag von Wilhelm Ernst & Sohn; Berlin.

Björling, P. R. and *Gissing, F. T.;* "Peat, Its Use and Manufacture"; 1907; Chas. Griffin & Co. Ltd.; London.

Brame, I. S. S.; "Fuel; Solid, Liquid and Gaseous"; 1924; Edward Arnold & Co.; London.

Cronshaw, H. B.; "Oil Shales"; 1921; John Murray; London.

Danger, L.; "Torfstreu und Torfmull"; 1901; Selbstverlag; Lübeck, Germany.

Davis, C. A.; "The Uses of Peat for Fuel and Other Purposes"; Bulletin No. 16; 1921; U. S. Bureau of Mines, Government Printing Office, Washington, D. C.

Dolch, Moritz; "Die rationelle Verwertung der niederwertigen Braunkohlen"; 1922; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Dubow, P. I. and *Tschelinzew, W. W.;* "Oelschiefer und ihre technische Verwertung" (in Russian); 1932; Onti WSNCH, SSSR; Leningrad.

Eckardt, Arthur; "Das Trocknen der Braunkohle und seine Wertschaftlichkeit"; 1913; Verlag von Wilhelm Knapp; Halle (Saale).

Erdmann, Ernst, "Die Chemie der Braunkohle"; 1st Edition 1907; *Erdmann, Ernst, and Doltch, Moritz,* 2nd Edition (Vol. III of "Die deutsche Braunkohlenindustrie") 1927; 3rd Edition 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Faber, Alfred; "Braunkohlengeneratorgas"; 1931; Verlag von R. Oldenbourg; Munich.

Fürth, Arthur; "Braunkohle und ihre chemische Verwertung" (Vol. XI of "Technische Fortschrittsberichte"); 1926; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Gavin, Martin J.; "Oil-Shale" (Bulletin 210); 1922; Bureau of Mines, Department of Interior; The Bradford-Robinson Printing Co.; Denver, Col.

George, R. D.; "Oil Shales of Colorado" (Bulletin No. 25); 1921; Colorado Geological Survey; Eames Brothers; Denver, Colorado.

Gissing, F. T.; "Commercial Peat, Its Uses and Possibilities"; 1909; Chas. Griffin & Co., Ltd.; London.

Gissing, F. T.; "Peat Industry Reference Book"; 1920; Chas. Griffin & Co., Ltd.; London.

Gothan, W. U. E. F.; "Studien über die Bildung der Schwelkohle und des Pyropissits"; 1925; Verlag von Wilhelm Knapp; Halle (Saale).

Graefe, Edmund; "Laboratoriumsbuch für die Braunkohlenteer-Industrie"; 1st Edition, 1908; 2nd Edition, 1923; Verlag von Wilhelm Knapp; Halle (Saale); 3rd Edition, 1931; Verlag von R. Oldenbourg; Munich.

Graefe, Edmund, "Die Braunkohlenteer-Industrie"; 1st Edition (Vol. II of Monographien über chemisch-technische Fabrikationsmethoden), 1906; 2nd Edition, 1922; Verlag von Wilhelm Knapp; Halle (Saale); 3rd Edition, 1931; Verlag von R. Oldenbourg; Munich.

Graefe, Edmund; "Industries des goudrons de lignite" (Translated from the German by Adolf Jouve); 1930; Ch. Béranger; Paris.

Grosse, W.; "Die volkwirtschaftliche Bedeutung der sächsische-thüringischen Braunkohlenteer- und Montanwachsindustrie"; 1923; Verlag von Wilhelm Knapp; Halle (Saale).

Halse, G. W.; "Oil and Retortable Materials" (A Handbook on the Utilization of Coal, Torbanite, Cannel and Oil Shale); 1927; Chas. Griffin & Co., Ltd.; London.

Hausding, Alfred; "Handbuch der Torfgewinnung und Torfverwertung"; 1st Edition, 1917; 2nd Edition, 1918; 3rd Edition, 1919; 4th Edition, 1920; 5th Edition, 1921; Verlag von Paul Parey; Berlin.

Hausding, Alfred; "A Handbook on the Winning and the Utilization of Peat" (Translated from the German); 1921; H. M. Stationery Office; London.

Hilliger, Bruno; "Die Trocknung und Schwelung der Braunkohle durch Spülgase"; 1926; Verlag von Julius Springer; Berlin.

Hilliger, Bruno and *Wurm;* "Untersuchungen zur Frage der Braunkohlenvergasung bei Gewinnung von Urteer"; 1921; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Höring, Paul; "Moornutzung und Torfverwertung"; 1915; Verlag von Julius Springer; Berlin.

Hradil, Guido and *Falser, Heinz v.;* "Die Oelschiefer Tirols"; 1930; Joh. Ambr. Barth; Leipzig.

Johnson, S. W.; "Peat and Its Uses"; 1886; Macmillan & Co., New York.

Kerr, W. A.; "Peat and Its Products"; 1905; Begg, Kennedy & Elder; Glasgow.

Koller, Theodor; "Die Torfindustrie"; 1898; A. Hartleben's Verlag; Vienna.

Klein, G.; "Handbuch für den deutschen Braunkohlenbergbau"; 3rd Edition (Vol. I of "Die deutsche Braunkohlenindustrie") 1927; Verlag von Wilhelm Knapp; Halle (Saale).

Klever, H. W. and *Mauch, Karl;* "Ueber den Esthländischen Oelschiefer Kukkersit" (Vol. 15 of Kohle-Koks-Teer); 1927; Verlag von Wilhelm Knapp; Halle (Saale).

Limberg, Th.; "Die Praxis des wirtschaftlichen Verschwelens und Vergasens"; 1925; Verlag von Wilhelm Knapp; Halle (Saale).

Luts, K.; "Der Estländische Brennschiefer-Kukersit, seine Chemie, Technologie und Analyse"; 1934; K. Mattiesens Buchdruckerei, Ant.-Ges.; Tartu, Estonia.

Marcotte, Edmond; "Les lignites et leurs applications industrielles"; 1925; Gauthier-Villars et Cie; Paris.

McKee, Ralph H.; "Shale Oil"; 1925; The Chemical Catalog Co., Inc.; New York.

de Mori, A.; "Carbonizzazione e distillazione del legno"; 1933; S. Lattes & Cie; Turin, Italy.

Potonié, Henry; "Die Enstehung der Braunkohlen und verwandten Bildungen einschlieszlich des Petroleums"; 1905; Gebrüder Bornträger; Berlin.

Potonié, Henry; "Die Entstehung der Steinkohle und der Kaustobiolithe überhaupt"; 1920; Gebrüder Bornträger; Berlin.

Potonié, Robert; "Allgemeine Petrographie der 'Oelschiefer' und ihrer Verwandten, mit Ausblicken auf die Erdölenstehung"; 1928; Gebrüder Bornträger; Berlin.

Richter, Carl; "Die mechanische Aufbereitung der Braunkohle"; 1st Edition 1910; *Richter, Carl* and *Horn, P.,* 2nd Edition (Vol. II of "Die deutsche Braunkohlenindustrie") 1926; Verlag von Wilhelm Knapp; Halle (Saale).

Salermo, Ltd.; "The Salermo Process and the Extraction of Oil from Oil Shales and Torbanites"; 1936; Salermo, Ltd.; London.

Sauer, A.; Grube, G.; von der Burchard, E.; and *Schmidt, Oskar.;* "Die Verwertung des Oelschiefers"; 1920; Konrad Wittwer Verlag; Stuttgart.

Scheithauer, Waldemar; "Die Fabrikation der Mineralöle und des Paraffins aus Schwelkohle, Schiefer, usw."; 1895; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Scheithauer, Waldemar; "Die Braunkohlenteerprodukte und das Oelgas"; 1st Edition, 1906; 2nd Edition, 1923; Verlag von Otto Spamer; Berlin.

Scheithauer, Waldemar; "Die Schwelteere, ihre Gewinnung und Verarbeitung"; 1st Edition, 1911; 2nd Edition, 1922; Verlag von Otto Spamer; Berlin.

Scheithauer, Waldemar; "Shale Oils and Tars and Their Products" (Translated from the German); 1st Edition, 1913; *Scheithauer, Waldemar* and *Stocks, H. B.;* 2nd Edition, Revised, 1923; Scott, Greenwood & Son; London.

Scheuer, Otto; "Gewinnung und Verwertung von Nebenerzeugnissen bei der Verwendung von Stein- und Braunkohlen"; 1915; F. C. Glaser; Berlin.

Schneider, Wilhelm; "Ueber Extraktion und Destillation der Braunkohle"; 1922; Verlag von Wilhelm Knapp; Halle (Saale).

Schulz, P.; "Des Probenehmen von Braunkohlen und die Untersuchung von Braunkohlenflözen"; 1921; Verlag von Wilhelm Knapp; Halle (Saale).

Stadnikov, G. L.; "Neuere Torfchemie"; 1930; Verlag von Theodor Steinkoff; Dresden and Leipzig.

Steinert, Johannes; "Torfveredelung"; 1926; Verlag von Wilhelm Knapp; Halle (Saale).

Steinert, Johannes; "Der Torf und seine Verarbeitung"; 1926; de Gruyter & Co.; Berlin.

Thau, Adolf; "Braunkohlenschwelöfen"; 1924; Verlag von Wilhelm Knapp; Halle (Saale).

Thau, Adolf; "Die Schwelung von Braun- und Steinkohle"; 1st Edition, 1928; Verlag von Wilhelm Knapp; Halle (Saale); 2nd Edition, 1931; Verlag von R. Oldenbourg; Munich.

Thenius, Georg; "Die Verwertung des Torfs und seiner Destillationsprodukte" (Vol. 280 of Chemtechn. Bibliothek); 1904; A. Hartleben's Verlag; Vienna.

von Winkler, Henry; Öpik, A.; Reinwaldt, J.; von Middendorff, K.; Ottoson, J. and *von Rennenkampff, W.;* "Der estländische Brennschiefer: Untersuchung, Gewinnung und Verwertung"; 1931; F. Wassermann; Revel.

Wölbling, Hans and *Hentze, E.;* "Oelschiefer, ihre Vorkommen, ihre Gewinnung und Verwendung"; 1928; Verlag von Wilhelm Knapp, Halle (Saale).

Würzner, E.; "Benzingewinnung aus Braunkohle"; 1932; Verlag von Wilhelm Knapp; Halle (Saale).

Zeidler, Rudolf; "Neue Wege der Verwertung des Oelschiefers und seine Umwandlung in Oele"; 1933; Kluge; Reval.

(E) COAL TARS AND PITCHES

Armstrong, John; "Carbonization Technology and Engineering"; 1929; J. B. Lippincott & Co.; Philadelphia.

Bauer, Julius; "Die Kohlen- und Oelhydrierung und ihrer heutigen Form, und die hier gegebenen Zusammenhänge technischer und wirtschaftlicher Natur"; 1932; Verlag Techn. Zeitschriften; Teplitz-Schönau.

Bunbury, H. M. and *David, A.;* "The Industrial Applications of Coal Tar Products"; 1925; Ernest Benn, Ltd.; London; and D. Van Nostrand Co., Inc.; New York.

Dolch, Moritz; "Der Drehrohrofen" (Sammlung Kohle-Koks-Teer); 1926; Verlag von Wilhelm Knapp; Halle (Saale).

Dolch, Moritz; "Brennstofftechnisches Praktikum"; 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Fischer, Franz; "Ueber die Mineralölgewinnung bei der Destillation und Vergassung der Kohlen"; 1920; Gebrüder Borntträger; Berlin.

Fischer, Franz; "Chemie der Kohle" (Vols. I and II); 1924; Gebrüder Borntträger; Berlin.

Fischer, Franz; "Umwandlung der Kohle in Oele"; 1925; Gebrüder Borntträger; Berlin.

Fischer, Franz and *Lessing, R.;* "The Conversion of Coal into Oils" (Translated from the German); 1925; D. Van Nostrand Co., Inc.; New York.

Galle, Ernst; "Hydrierung der Kohle, Teere und Mineralöle" (Vol. XXVII of "Technische Fortschrittsberichte. Fortschritte der chemische Technologie in Einzeldarstellungen"); 1932; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Gentry, F. M.; "The Technology of Low Temperature Carbonization"; 1928; The Williams & Wilkins Co.; Baltimore, Md.

Gluud, Wilhelm; "Die Tieftemperaturverkokung der Steinkohle"; 1st Edition, 1919; 2nd Edition, 1921; Verlag von Wilhelm Knapp; Halle (Saale).

Gluud, Wilhelm and *Jacobson, D. L.;* "International Hand-Book of the By-Product Coke Industry" (Based on revised German edition); 1932; The Chemical Catalog Company, Inc., New York; and Wilhelm Knapp; Halle (Saale).

Gluud, Wilhelm (also *G. Schneider* and *Heinrich Winter*); "Handbuch der Kokerei" (Vols. I and II); 1927; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Heinrichsen, F. W. and *Taczaks;* "Die Chemie der Steinkohle"; 1916; Verlag von Wilhelm Engelmann; Leipzig.

Heydenreich, F. A.; "Die deutsche Steinkohlenindustrie und ihre wirtschaftlichen Zusammenhänge"; 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Jacobson, D. L.; "Gluud's International Handbook of the By-Product Coke Industry"; 1932; The Chemical Catalog Co., Inc.; New York.

Keller, Konrad; "Laboratoriumsbuch für die Kokerei- und Teerproduktenindustrie der Steinkohle"; 1st Edition, 1922; 2nd Edition, 1923; Verlag von Wilhelm Knapp; Halle (Saale).

Kraemer, G. and *Spilker, Adolf;* "Steinkohlenteer"; 1909; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Lander, C. H. and *McKay;* "Low Temperature Carbonization"; 1924; Ernest Benn, Ltd.; London.

Litinsky, L.; "Kokerei und Gaswerksöfen" (Vol. 17 of "Kohle, Koks und Teer", by *J. Gwosdz*); 1930; Verlag von Wilhelm Knapp; Halle (Saale).

Lunge, Georg; "Die Industrie des Steinkohlenteers und des Ammoniaks"; 1st Edition, 1883; 2nd Edition, 1886; 3rd Edition, 1888; *Lunge, Georg* and *Köhler, Hippolyt;* 4th Edition (2 Vols.), 1900; 5th Edition (2 Vols.), 1912; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Lunge, Georg; "Coal Tar and Ammonia" (Translated from the German); 1st Edition, 1882; 2nd Edition, 1887; 3rd Edition, 1893; 4th Edition (2 Vols.), 1909; D. Van Nostrand Co., New York; 5th Edition (2 Vols.), 1916; Gurney & Jackson; London.

Malatesta, Guiseppe, "Il catrame ed i suoi derivati"; 1918; Ulrico Hoepli; Milan.

Malatesta, Guiseppe; "Le Goudron et ses Dérivés" (Translated from the Italian by J. A. Montpellier); 1st Edition, 1918; 2nd Edition, 1927; P. Vicq-Dunod et Cie; Paris.

Malatesta, Guiseppe; "Coal Tars and Their Derivatives" (Translated from the Italian); 1920; E. & F. N. Spon, Ltd.; London.

Meade, Alwyne and *Jones, S. H.;* "Modern Gas-Works Practice"; 2nd Edition, 1921; D. Van Nostrand Co. Inc.; New York.

North, Sydney and *Garbe, J. B.;* "Low Temperature Distillation"; 1925; Isaac Pitman & Sons; New York.

Odell, W. W.; "Preparation and Uses of Tar and Its Simple Crude Derivatives" (Tech. Paper No. 268); 1922; Department of Interior, Bureau of Mines, U. S. Government Printing Office; Washington, D. C.

Oppler, Theodor; "Handbuch der Fabrikation mineralischer Oele aus Steinkohlen, Braunkohlen, Holz, Torf, Petroleum, und anderen bituminösen Substanzen"; 1862; Verlag von Julius Springer; Berlin.

Porter, Horace C.; "Coal Carbonization"; 1924; The Chemical Catalog Co. Inc.; New York.

Roberts, John; "Coal Carbonization"; 1927; Isaac Pitman & Sons; New York.

Rosendahl, Fritz; "Steinkohlenteer" (Vol. XXXII of Technische Fortschrittsberichte); 1934; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Russell, W. M.; "Operation of Gas Works"; 1st Edition; 1917; D. Van Nostrand Co. Inc.; New York.

Schreiber, Fritz; "Industrie der Steinkohlenveredelung"; 1923; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Schultz, Gustav T. A. O.; "Die Chemie des Steinkohlenteers"; 1st Edition, 1882; 2nd Edition, 1886; 3rd Edition, 1888; 4th Edition, 1890; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Spielmann, P. E.; "The Constituents of Coal-Tar"; 1924; Longmans, Green & Co., Ltd.; London.

Spilker, Adolf and *Weissgerber, R.;* "Steinkohlenteer"; 1917; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Spilker, Adolf, "Kokerei und Teerprodukte der Steinkohle"; 1st Edition, 1908; *Spilker, Adolf; Dittmer, O.;* and *Weissgerber, R.;* 4th Edition, 1923; 4th Edition, Revised, 1931; *Spilker, Adolf; Dittmer, O.;* and *Kruber, O.;* 5th Edition, 1933; Verlag von Wilhelm Knapp; Halle (Saale).

Strache, H. and *Lant, R.;* "Kohlenchemie"; 1924; Akadem. Verlagsgesellschaft m.b.H.; Leipzig.

Strache, H. and *Ulmann, H.;* "Leitfaden der Technologie der Brennstoffe"; 1927; F. Deuticke; Leipzig.

Thau, Adolf; "Die Schwelung von Braun- und Steinkohle"; 1st Edition, 1930; Verlag von Wilhelm Knapp; Halle (Saale); 2nd Edition, 1931; Verlag von R. Oldenbourg; Munich.

Thenius, Georg; "Die technische Verwertung des Steinkohlenteers"; 2nd Edition, 1878; 3rd Edition, 1899; A. Hartleben's Verlag; Vienna, Budapest & Leipzig.

Vulitch, Vladimir de.; "Les produits industriels des goudrons de houilles et leurs applications"; 1908; Gauthier-Villars et Cie; Paris.

Wagner, F. H.; "Coal Gas Residuals"; 1st Edition, 1914; McGraw-Hill Book Co., Inc.; New York.

Wagner, F. H.; "Coal and Coke"; 1st Edition, 1916; McGraw-Hill Book Co. Inc.; New York.

Warnes, Arthur R.; "Coal Tar Distillation"; 1st Edition, 1914; 2nd Edition, 1918; 3rd Edition, 1924; Benn Brothers Ltd.; London.

Weiss, J. M.; "Methods of Analysis of the Coal-Tar Industry"; 1919; The Barrett Co.; New York.

Weissgerber, R.; "Chemische Technologie des Steinkohlenteers"; 1923; Verlag von Otto Spamer; Leipzig.

Wellington, S. N. and *Cooper, W. R.;* "Low Temperature Carbonization"; 1928; J. B. Lippincott & Co.; Philadelphia.

Winter, Heinrich; "Taschenbuch für Gasanstalten, Kokereien, Schwelereien und Teerdestillationen"; 2nd Edition, 1927; 3rd Edition, 1928; 4th Edition, 1929; 5th Edition, 1930; Verlag von Wilhelm Knapp; Halle (Saale).

Winter, Heinrich; "Brennstoffe, ihre Verwertung und ihre Veredelung"; 1930; Verlag von Wilhelm Knapp; Halle (Saale).

Wirth, F.; "Brennstoffchemie"; 1922; G. Stilke; Berlin.

Wölbling, Hans; "Die Hydrierung, mit besonderer Berücksichtigung der Brennstoffe und ihre Destillationsprodukte"; 1926; Verlag von Wilhelm Knapp; Halle (Saale).

(F) PETROLEUM PRODUCTS

Aisinman, S.; "Taschenbuch für die Mineralöl-Industrie" ("Die einheil. Prüfungsmethoden in die Mineralöl-Industrie"); 1896; Verlag von Julius Springer; Berlin.

Aisinman, S.; "Die destruktive Destillation in der Erdöl-Industrie" (Sammlung chemischer und chemisch-technischer Vorträge, Band V, Heft 6); 1900; Verlag von Ferdinand Enke; Stuttgart.

Albrecht, A.; "Das Paraffin und die Mineralöle"; 1874; E. Koch; Stuttgart.

Albrecht, M. u. E.; "Das Erdöl und seine Produkte"; 1909; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Aldebert, M. P.; "Situation pétrolifère actuelle au Caucase"; 1920; Ch. Béranger; Paris.

American Institute of Mining and Metallurgical Engineers; "Petroleum Development and Technology in 1927"; Published 1928; Same in 1928-1929, Published 1929; Same in 1930, Published 1930; Same in 1931, Published 1932; Same in 1932, Published 1933; American Institute of Mining and Metallurgical Engineers; New York.

Andros, S. O.; "The Petroleum Handbook"; 1919; The Shaw Publishing Co.; Chicago.

Anon; "Petroleum Engineering Handbook"; 2nd Edition, 1931; Palmer Publications; Los Angeles, Cal.

Antisell, Thomas; "The Manufacture of Photogenic Hydrocarbon Oils"; 1859; D. Appleton & Co.; New York.

Arends, H. and *Morsner, C.;* "Handbuch für die internationale Petroleum-Industrie für 1911"; Published 1911; Same for 1912, Published 1912; Same for 1913, Published 1913; Same for 1914, Published 1914; Same for 1927-28, Published 1929; *Arends, H.* and *Tokayer, O.;* Same for 1929, Published 1929; Finanzverlag, G.m.b.H.; Berlin.

Arnold, Ralph and *Darnell, J. L.;* "Manual for the Oil and Gas Industry"; 1920; John Wiley & Sons, Inc.; New York.

Arnold, Ralph and *Kemnitzer, W. J.;* "Petroleum in the United States and Possessions"; 1931; Harper Bros.; New York.

Bacon, R. F. and *Hamor, W. A.;* "The American Petroleum Industry" (Vols. I and II); 1916; McGraw-Hill Book Co., Inc.; New York.

Baron, C.; "Au pays de l'or noir (Bakou, Grozney). Le pétrole russe"; 1935; Ch. Béranger; Paris.

Battle, J. R.; "The Handbook of Industrial Oil Engineering"; 1st Edition, 1920; 2nd Edition, 1923; 3rd Edition, 1928; 4th Edition, 1931; J. B. Lippincott & Co.; Philadelphia.

Bell, E. C.; "History of Petroleum"; 1900; The Bugle Print; Titusville, Pa.

Bell, H. S.; "American Petroleum Refining"; 1st Edition, 1923; 2nd Edition, 1929; D. Van Nostrand Co. Inc.; New York.

Bentz, Herrmann and *Krais, Stutzer;* "Deutsches Erdöl"; 1931; Verlag von Ferdinand Enke; Stuttgart.

Bérenger, Henry; "Le pétrole et la France"; 1920; E. Flammarion; Paris.

Blum, Anthony; "Petroleum: Where and How to Find It"; 1922; Modern Mining Books Publishing Co.; Chicago.

Blumer, Ernst; "Die Erdöllagerstätten und übriges Kohlenwasserstoffvorkommen der Erdrinde"; 1922; Verlag von Ferdinand Enke; Stuttgart.

Boileau, Gauldrée; "Exploitation de l'huile minérale dans "Amérique du Nord"; 1863; H. Dunod et Cie.; Paris.

Bonand, R. de.; "Le pétrole, origine, gisements, commerce, succédanés"; 1920; Ch. Béranger; Paris and Liege.

Bone, J. H. A.; "Petroleum and Petroleum Wells"; 1865; J. B. Lippincott & Co.; Philadelphia.

Bowles, C. E.; "The Petroleum Industry"; 1922; Schooley Stationery & Printing Co.; Kansas City, Mo.

Boyd, R. N.; "Petroleum, its Development and Uses"; 1895; Whittaker & Co.; London.

Brannt, W. T.; "Petroleum; its History, Origin, Occurrence Production, Physical and Chemical Constitution, Technology, Examination and Uses" (Based on German publication by Hans Höfer and Alexander Veith); 1895; Henry Cary Baird & Co.; Philadelphia; Sampson Low, Marston & Co., Ltd.; London.

Brooks, B. T.; "Non-Benzenoid Hydrocarbons"; 1922; The Chemical Catalog Co., Inc.; New York.

Buchner, O.; "Die Mineralöle, inbesondere Photogen, Solaröl, und Petroleum"; 1864; Varlag von Bernhard Friedrich Voigt; Weimar and Leipzig.

Budowski, J.; "Die Naphtensaüren"; 1922; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; also Verlag von Julius Springer; Berlin.

Burgmann, Arthur; "Petroleum und Erdwachs" (Vol. 72 of Hartleben's Bibliothek); 1880; A. Hartleben's Verlag; Vienna.

Burstin, Hugo; "Untersuchungsmethoden der Erdöl-industrie"; 1930; Verlag von Julius Springer; Berlin.

Compbell, Andrew, "Petroleum Refining"; 1st Edition, 1918; Charles Griffin & Co., London; 2nd Edition, 1922; Petroleum Age, New York; 3rd Edition, 1929; J. B. Lippincott & Co., Philadelphia.

Chartrou, J. J.; "Pétroles naturels et artificiels"; 1931; Armand Colin; Paris.

Chautard, J.; "Les gisements de pétrole"; 1922; Gaston Doin; Paris.

Clapp, F. G.; "Petroleum and Natural Gas Resources of Canada" (Vols. I and II); 1914; Government Printing Bureau; Ottawa.

Cogniet, Charles; "Les huiles minérales au point de vue de leur emploi pour le chauffage des machines à vapeur"; 1868; E. Lacroix; Paris.
Colomer, Félix and *Lordier, C.;* "Combustibles industriels; houille, pétrol, lignite; tourbe, bois, charbon de bois, agglomérés, coke, gaz, résidus industriels ou agricoles, gadoues"; 1919; H. Dunod and E. Pinat; Paris.
Colomo, Jose and *Ortega, Gustave;* "La industria des petróleo en México; 1928; Talleres Gráficos de la Nación: Mexico City.
Cone, Andrew and *Johns, W. R.;* "Petrolia; A Brief History of the Pennsylvania Petroleum Region"; 1870; D. Appleton & Co.; New York.
Courau, Robert; "Technique des pétroles"; 1921; Gaston Doin; Paris.
Cox, G. H., Dake, C. L. and *Muilenburg, G. A.;* "Field Methods in Petroleum Geology"; 1921; McGraw-Hill Book Co., Inc.; New York.
Crew, Benjamin J.; "A Practical Treatise on Petroleum"; 1887; C. A. Ashburner; Philadelphia.

Dalloni, M.; "La géologie du pétrole et la recherche des gisements pétrolifères en Algérie"; 1923; J. Carbonel; Alger.
Dauthcombe, J. E.; "Chemistry of the Oil Industries"; 1914; D. Van Nostrand Co. Inc.; New York.
Day, D. T.; "Handbook of the Petroleum Industry" (Vols. I and II); 1922; John Wiley & Sons, Inc.; New York.
Delaisi, Francis; "Le pétrole"; 1921; Librairie Payot et Cie.; Paris.
Delehaye, H.; "Huiles minérales"; 1911; Ch. Béranger; Paris.
Deutsch, Henry; "Le pétrole et ses applications"; 1st Edition, 1891; 2nd Edition, 1906; Librairies-Imprimeries Réunies; Paris.
Donahue, D. D.; "A Treatise on Petroleum and Natural and Manufactured Gases"; 1902; Pantagraph Print & Stationery Co.; Bloomington, Ill.
Donath, Edward and *Lissner, A.;* "Kohle und Erdöl" (Sonderausgabe aus der Sammlung chemischer und chemisch-technischer Vorträge, Band 26); 1920; Verlag von Ferdinand Enke; Stuttgart.
Dücker, F. F. v.; "Petroleum und Asphalt in Deutschland"; 1881; Verlag von J. C. C. Bruns; Minden.
Dunstan, A. E.; "Petroleum Industry"; 1922; Institution of Petroleum Technologists; London.
Dunstan, A. E.; "Petroleum: Twenty-Five Years' Retrospect, 1910-1935"; 1935; Institution of Petroleum Technologists; London.
Dunstan, A. E.; "Petroleum Technology 1935"; 1936; Institution of Petroleum Technologists; London.
Dunstan, A. E. and *Sell, George;* "Proceedings of the World Petroleum Congress, London" (Vols. I and II); 1934; World Petroleum Congress; Aldine House, Bedford Street; London.
Dupaigne, Albert; "Le pétrole, son histoire, sa nature, ses usages et ses dangers"; 1872; Palmé; Paris.
Duque, Rufino; "El Petróleo"; 1924; Artes de la Ilustración; Madrid.

Egloff, Gustav; "Earth Oil"; 1933; The William & Wilkins Company; Baltimore, Md.
Egloff, Gustav; Levinson, B. L.; and *Herrman, M.;* "The Cracking Art in 1930-1931"; 1932; *Egloff, Gustav, Crandal, E. E.,* and *Doty, M. M.;* "The Cracking Art in 1935"; 1936; Universal Oil Products Co.; Chicago, Ill.

Ehrsam, R.; "Fabrication des huiles minérales et pyrogénées"; 1909; Published by the author; Charenton-le-Point, Seine.

Eichwald, Egon; "Mineralöle" (Vol. VII of "Fortschritte der chemische Technologie in Einzeldarstellungen"); 1925; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Ellis, Carleton; "The Chemistry of Petroleum Derivatives"; 1934; The Chemical Catalog Co., Inc.; New York.

Emmons, W. H.; "Geology of Petroleum"; 1921; McGraw-Hill Book Co., Inc.; New York.

Engelder, Carl J.; "Laboratory Manual of Gas, Oil and Fuel Analysis"; 1931; John Wiley & Sons, Inc.; New York.

Engler, Carl; "Die deutschen Erdöle"; 1887; Verhandlung des Vereins für Gewerbefleisz, Verlag von L. Simion; Berlin.

Engler, Carl; "Die neueren Ansichten über die Enstehung des Erdöles"; 1907; Verlag für Fachliteratur, G.m.b.H.; Berlin.

Engler, Carl, and *Höfer, Hans,* "Das Erdöl"; Vol. I, 1913; Vol. II, 1909; Vol. III, 1911; Vol. III (2nd Edition, Revised by *Jenoe Tausz*), 1930; Vol. IV, 1916; Vol. IV (2nd Edition, Revised by *Jenoe Tausz*), 1930; Vol. V, 1919; Vol. VI, 1925; S. Hirzel Verlag; Leipzig.

Estrada, Genaro; "Bibliografica del petroleo en Mexico" (Monografias Bibliograficas Mexicanas Numero 8); 1927; Subsecretario de Relaciones Exteriores; Mexico City.

Evrard and *Dislère;* "Sur les huiles de pétrole" (Mémoires de la Société d'agriculture, de sciences et d'arts de Douai); 1866; Crépin; Douai.

Faber, Alfred; "Die neueste Entwicklung der Welterdölwirtschaft und die Mineralöllage Deutschlands"; 1926; Verlag von Wilhelm Knapp; Halle (Saale).

Fabris, Guido; "Olii e Grassi: Vegetali, Animali e Minerali"; 2nd Edition, 1923; Ulrico Hoepli; Milan.

Farez, Ingénieur; "Quelques traits de l'histoire du pétrole, son origine et celle de la houille"; 1875; Crépin; Douai.

Filhol, J. and *Bihoreau, Ch.;* "Le pétrole"; 1929; Editions pittoresques; Paris.

Forbin, Victor; "Ce qu'il faut connaître du pétrole"; 1937; Boivin & Cie; Paris.

Fussteig, R.; "Theorie und Technik des Krackens"; 1936; Allgemeiner Industrie-Verlag G.m.b.H.; Berlin-Lichterfelde.

Garfias, V. R.; "Petroleum Resources of the World"; 1923; John Wiley & Sons, Inc.; New York; Chapman & Hall; London.

Gehrcke, Franz; "Petroleumhandel in Deutschland"; 1906; Lauppe; Tübingen.

Glangeaud, P.; "Note sur les recherches de pétrole en Limagne"; 1924; J. Hermann; Paris.

Graetz, A.; "Pétroles naturels et carburants de synthèse"; 1931; J. B. Baillière et Fils; Paris.

Grafe, Victor; "Warenkunde und Technologie der Konservierungsverfahren der Kohle und des Erdöls"; 1928; C. E. Pöschel Verlag; Stuttgart.

Greene, J. Arthur; "A Treatise on British Mineral Oil"; 1920; Charles Griffin & Co., Ltd.; London.

Gruse, W. A.; "Petroleum and its Products"; 1928; McGraw-Hill Book Co., Inc.; New York.

Gubkin, J. M.; "Lehre von Erdöl"; 1932; Wissenschaftl.-Techn. Erdöl-Verlag; Moscow.

Guiselin, A.; "Les combustibles liquides, essences, huiles, résidus de pétroles, de goudrons de houille, de schistes bitumineux, de lignites, de tourbe, de bois, etc."; 1920; H. Dunod & E. Pinat; Paris.

Gürich, G.; "Das Erdöl in Nordwestdeutschland"; 1917; L. Friederichsen und Co.; Hamburg.

Gurwitsch, Leo; "Wissenschaftliche Grundlagen der Erdöl-Verarbeitung"; 1st Edition, 1913; 2nd Edition, 1924; 3rd Edition, 1929; Verlag von Julius Springer; Berlin.

Gurwitsch, Leo; "Traitement des huiles minérales" (Translated from the German by Marcel Thiers); 1925; Ch. Béranger; Paris.

Gurwitsch, Leo and *Moore, Harold;* "The Scientific Principles of Petroleum Technology" (Translated from the German); 1st Edition, 1926; 2nd Edition, 1932; Chapman & Hall, Ltd.; London.

Guttentag, W. E.; "Petrol and Petroleum Spirits, Sources, Preparation, Examination and Uses"; 1919; Longmans, Green & Co., Ltd.; London and New York.

Hager, Dorsey; "Practical Oil Geology"; 1915; McGraw-Hill Book Co., Inc.; New York.

Hager, Dorsey; "Oil-Field Practice"; 1921; McGraw-Hill Book Co., Inc.; New York.

Hamor, W. A. and *Padgett, F. W.;* "The Technical Examination of Crude Petroleum and Petroleum Products"; 1920; McGraw-Hill Book Co., Inc.; New York.

Handel, J. A.; "Recherche et exploitation du pétrole"; 1923; H. Dunod et Cie.; Paris.

Haudoin, Hippolyte and Soulié, Émile; "Le pétrole, ses gisements, son exploitation, son traitement industriel, ses produits dérivés, ses applications à l'éclairage et au chauffage"; 1864; Eugene Lacroix; Paris.

Hecht, F. E.; "Erdöl-Muttersubstanz"; 1935; Verlag von Ferdinand Enke; Stuttgart.

Heck, F.; "Die deutsche Erdölindustrie"; 1908; Aachener Verlags- und Druckerei-Gesellschaft; Aachen.

Henry, J. D.; "Oil Fields of the Empire"; 1910; Bradbury, Agnew & Co., Ltd.; London.

Henry, J. D.; "History and Romance of the Petroleum Industry" (Vols. I, II and III); 1914-1915; Bradbury, Agnew & Co., Ltd.; London.

Henry, J. G.; "The Early and Later History of Petroleum"; 1873; Jas. B. Rodgers & Co.; Philadelphia.

Hicks, J. A.; "Laboratory Handbook of Mineral Oil Testing"; 1st Edition, 1906; 2nd Edition, 1912; 3rd Edition, 1919; 4th Edition, 1926; Charles Griffin & Co., Ltd.; London.

Höfer, Hans; "Die Petroleum Industrie Nord-Amerikas in geschichtlicher, wirtshaftlicher, geologischer und technischer Hinsicht" ("Bericht über die Weltausstellung in Philadelphia 1876", Heft 8); 1877; Der Oesterreich. Commission für die Weltanstellung in Philadelphia, 1876; Vienna.

Höfer, Hans; "Das Erdöl und seine Verwandten"; 1st Edition, 1888; 2nd Edi-

tion, 1900; 3rd Edition, 1912; 4th Edition, 1922; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Höfer, Hans; "The Geology, Extraction and Transportation of Petroleum"; 1909; S. Hirzel Verlag; Leipzig.

Höfer, Hans; "Die Nomenklatur in der Erdölwissenschaft"; 1915; Verlag für Fachliteratur, G.m.b.H.; Berlin.

Holde, David; "Untersuchung der Schmiermittel"; 1st Edition, 1897; "Untersuchung der Mineralöle und Fette"; 2nd Edition, 1905; 3rd Edition, 1909; "Untersuchung der Kohlenwasserstofföle und Fette"; 4th Edition, 1913; *Holde, David* and *Meyerhein, G.;* 5th Edition, 1918; "Kohlenwasserstofföle und Fette"; 6th Edition, 1924; *Holde, David,* and *Bleyberg, W.;* 7th Edition, 1933; Verlag von Julius Springer; Berlin.

Holde, David; "Traité d'analyse des huiles minérales" (Translated from the German by M. Gautier); 1909; Ch. Béranger; Paris.

Holde, David; "Huiles et grasses minérales, végétables et animales" (Translated from the German by Adolf Jouve); 1929; Ch. Béranger; Paris.

Holde, David, and *Mueller, Edward;* "The Examination of Hydrocarbon Oils and Saponifiable Fats and Waxes"; 1st Edition (Translated from the 4th German Edition), 1915; 2nd Edition (Translated from the 5th German Edition), 1922; John Wiley & Sons, Inc.; New York.

Hopkins, Erastus; "The Oil Chemists' Handbook"; 1900; John Wiley & Sons, Inc.; New York.

Hue, Fernand; "Le pétrole, son histoire, ses origines, son exploitation dans tous les pays due monde"; 1885; Lecène et Oudin; Paris.

Hunt, T. S.; "Notes on the History of Petroleum or Rock Oil"; 1862; G. E. Desbarats; Quebec.

Hunt, T. S.; "Petroleum; its Geological Relations considered with special Reference to its Occurrence in Gaspé"; 1865; G. E. Desbarats; Quebec.

Hurlburt, E. N.; "Mineral Oils, Tables, etc." 1918; Taylor Instrument Co.; Rochester, N. Y.

Independent Oil Mens' Association; "Petroleum Annual 1921"; Independent Oil Mens' Association; Chicago, Ill.

Institution of Petroleum Technologists; "Standard Methods for Testing Petroleum and its Products"; 1st Edition, 1924; 2nd Edition, 1929; 3rd Edition, 1935; 4th Edition, 1936; W. Speaight & Sons, Ltd., Institution of Petroleum Technologists; London.

Institution of Petroleum Technologists; "Reports on the Progress of Naphthology"; 1932; Aldine House, Bedford Street, Strand, London.

Jauch, Louis; "Le pétrole et son industrie"; 1921; Augustin Challomel; Paris.

Kalichevsky, V. A. and *Stagner, B. A.;* "Chemical Refining of Petroleum" (A. C. S. Monograph No. 63); 1933; The Chemical Catalog Co., Inc.; New York.

Kewley, James; "The Petroleum and Allied Industries: Petroleum, Natural Gas, Natural Waxes, Asphalts and Allied Substances"; 1922; Baillière, Tindall & Cox; London; D. Van Nostrand Co. Inc.; New York.

Killebrew, J. B.; "The Oil Region of Tennessee"; 1877; The American Printing Co.; Nashville, Tenn.

Kissling, Richard; "Laboratoriumsbuch für die Erdölindustrie"; 1st Edition, 1908; 2nd Edition, 1923; Verlag von Wilhelm Knapp; Halle (Saale).

Kissling, Richard; "Chemische Technologie des Erdöls und der ihm nahestehende Naturerzeugnisse: Erdgas, Erdwachs und Erdpech (Asphalt)"; 1st Edition, 1915; 2nd Edition, 1924; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Kissling, Richard; "Das Erdöl, seine Verarbeitung und Verwendung" (Vol. 12 of Monographien über Chemisch-technische Fabrikationsmethoden); 1st Edition, 1908; 2nd Edition, 1922; Verlag von Wilhelm Knapp; Halle (Saale).

Kissling, Richard; "Das Erdöl" (Vol. V of Monographien aus dem Gebiete der Fett-Chemie); 1923; Wissenschaftliche Verlagsgesellschaft m.b.H.; Stuttgart.

Kissling, Richard; "Die Mineralöle, ihre Gewinnung und Verwertung"; 1924; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; Walter de Gruyter & Co.; Berlin and Leipzig.

Kissling, Richard; "Industrie du pétrole" (Translated from the German by Ad. Jouve); 1924; Ch. Béranger; Paris.

Koetschau, Rudolf; "Erdöl und verwandte Stoffe" (Sonderausgabe aus Doelter-Leitmeier "Handbuch des Mineralchemie"); 1929; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Koettnitz, C.; "Allgemeine Erdölkunde für Industrie und Handel"; 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Kolbe, Ernest Aug.; "Die staatliche Mineralölfabrik Drohobycz"; 1920; Druck und Verlag der k. k. Hof- und Staatsdruckerei; Vienna.

Kraemer, A. J. and *Calkin, L. P.;* "Properties of Typical Crude Oils from the Producing Fields of the Western Hemisphere"; 1925; Government Printing Office, Department of Commerce; Washington, D. C.

Krämer, G. and *Böttcher;* "Die deutschen Erdöle und deren Verbreitung"; 1887; Verhandlung des Vereins für Gewerbefleisz; Verlag von L. Simion; Berlin.

Krejci-Graf, K.; "Die rumänischen Erdöllagerstatten"; 1929; Verlag von Ferdinand Enke; Stuttgart.

Krüger, Karl; "Oelvorkommen, Oelförderung, Oelschiefer, Oelpolitik"; 1924; E. Schweizerbart'sche Verlagsbuchhandlung (Erwin Nägele) G.m.b.H.; Stuttgart.

Krüger, Karl and *Poschardt, G. R.;* "Die Erdöl-Wirtschaft der Welt"; 1926; E. Schweizerbart'sche Verlagsbuchhandlung (Erwin Nägele) G.m.b.H.; Stuttgart.

Kuhn, Robert; "Mineralöltechnik"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Kwjatkowsky, N. A.; "Anleitung zur Verarbeitung von Naphtha und ihrer Produkte"; 1904; Verlag von Julius Springer; Berlin.

Lecomte-Denis, Maurice; "Le pétrole en France"; 1924; H. Dunod et Cie.; Paris.

Leet, A. N.; "Petroleum Distillation"; 1884; Oil, Paint and Drug Publishing Co.; New York.

Legendre, Jules; "Les pétroles Polonais"; 1923; Revue Petrolifère; Paris.

Lepoutre, Jean; "Le pétrole, facteur economique mondial"; 1923; Impr. nationale des mutilés; Liège.

Levi, Georges; "Éléments de la technique du pétrole"; 1926; L'Université de Strasbourg, l'Institut du Pétrole; Paris.

Lidgett, Albert; "Petroleum"; 1st Edition, 1919; 2nd Edition, 1928; 3rd Edition, 1929; Sir Isaac Pitman & Sons, Ltd.; London and New York.

Lilley, E. R.; "The Oil Industry"; 1925; D. Van Nostrand Co. Inc.; New York.

Macfarlane, J. M.; "Fishes, the Source of Petroleum"; 1924; The Macmillan Co.; New York.

Magnier, Désiré; "Nouveau manuel complet de la formation et de l'emploi des huiles minérales"; 1867; Librairie Roret; Paris.

Martin, A.; "Le pétrole, son origin, sa composition, sa recherche"; 1923; Felix Alean; Paris.

Martius, C. A.; "Die amerikanische Petroleum-Industrie"; 1877; Verlag von Julius Springer; Berlin.

Marvin, Charles; "The Petroleum of the Future; Baku, the Petrolia of Europe"; 1884; R. Andersen; London.

Marvin, Charles; "The Region of the Eternal Fire; An Account of a Journey to the Petroleum Region of the Caspian in 1883"; 1st Edition, 1884; 2nd Edition, 1891; W. H. Allen & Co., Ltd.; London.

Marvin, Charles; "The Petroleum Industry of Southern Russia"; 1884; Sonnenschein (Swan) & Co. Ltd.; London.

Marvin, Charles; "The Coming Oil Age; Petroleum—Past, Present and Future"; 1889; W. H. Allen & Co. Ltd.; London.

Masméjean, A. and *Béréhare, E.;* "Le pétrole, son utilisation comme combustible"; 1920; H. Dunod et Cie; Paris.

Mayer, A. W. J.; "De Destillatie van Mexicaansche Aaadolie"; 1932; Naamlooze Vennootschap W. D. Meinema; Delft, Holland.

Mendel, Joseph; "Die Entwicklung der internationalen Erdölwirtschaft"; 1922; K. F. Köhler; Leipzig.

Mesa y Ramos, José; "Pozos artesianos y porzos petróleo"; 3rd Edition, 1925; Romo; Madrid.

Meszmer, Hermann; "Das Erdöl, die Erdölindustrien und deren Erzeugnisse"; 1913; Verlag Jos. Kösel; Kempten.

Mills, E. J.; "Destructive Distillation; A Manualette of the Paraffin, Coal-Tar, Rosin-Oil, Petroleum and Kindred Industries"; 1st Edition, 1877; 2nd Edition, 1882; John Van Voorst, London; 3rd Edition, 1887; J. & A. Churchill, London; 4th Edition, 1892; Gurney & Jackson, London.

Mitchell, C. A.; "Oil: Animal, Vegetable, Essential and Mineral"; 1910; Sir Isaac Pitman & Sons, Ltd.; London.

Mitzakis, Marcel; "The Russian Oil Fields and Petroleum Industry"; 1912; Chapman & Hall, Ltd.; London.

Mitzakis, Marcel; "Oil Encyclopedia"; 1922; Chapman & Hall, Ltd.; London.

Möller, F. W.; "Die Versorgung der Welt mit Petroleum, mit besonderer Berücksichtigung Deutschlands"; 1910; Verlag für Fachliteratur, G.m.b.H.; Berlin.

Monti, Mario; "Il Mercato Mondiale del Petrolio, dal 1900 ad Oggi" (No. 4); 1922; Industria Tipografica Romana, via Ennio Quirino Visconti; Rome.

Montgruel, L. P.; "Traité pratique industriel et commercial des huiles minérales à l'usage des fabricants, marchands et consommateurs de pétroles, schistes et autres huiles analogues"; 1864; Gauthiers-Villars et Cie.; Paris.

Mosler, Chr.; "Die Petroleum-Industrie der Vereinigten Staaten von Nord-Amerika im Jahr 1876"; 1877; Verlag von Wilhelm Knapp; Halle (Saale).
Mossner, Julius; "Handbuch der internationalen Petroleum-Industrie, 1934"; 1935; "Die deutsche Kraftstoffwirtschaft"; 1937; Finanz-Verlag, G.m.b.H.; Berlin.
Murray, John; "Petroleum"; 1921; John Murray; London.

Nabuco de Araujo Jor., C. E.; "Petroleo"; 1936; J. R. Oliveira & Co.; Rio de Janeiro.
Naphtali, M.; "Leichte Kohlenwasserstofföle (Patentliteratur 1921-1927)"; 1928; Verlag M. Krayn; Berlin.
Naphtali, M.; "Naphthensäuren und Naphthensulfosäuren (Nachtrag 1927-1933, zu "Chemie, Technologie und Analyse der Naphthensäuren)"; 1934; Wissenschaftliche Verlagsgesellschaft, m.b.H.; Stuttgart.
Nauwelaerts, L.; "Petroleum"; 1936; N. V. Het Nederlandsche Boekhuis; Tilburg;
Nelson, W. L.; "Petroleum Refinery Engineering"; 1st Edition, 1936; McGraw-Hill Book Co., Inc.; New York City.
Neuburger, Henry and *Noalhat, Henri;* "Technologie du pétrole"; 1900; J. Fritsch; Paris.
Neuburger, Henry and *Noalhat, Henri;* "Technology of Petroleum; the Oil Fields of the World, their History, Geography and Geology, etc." (Translated from the French by J. G. McIntosh); 1901; Scott, Greenwood & Co.; London.
Neuendahl, L. v.; "Des Vorkommen des Petroleums in Galizien und dessen Gewinnung"; 1865; ———; Vienna.
Nicolescu, C. P.; "Gisements pétrolifères de l'Irak"; 1934; Les Presses Modernes; Paris.
Nimptsch, Reinhold; "Beiträge zum russischen Erdölproblem"; 1926; H. Sack Verlag; Berlin.
North, S. H.; "Petroleum"; 1904; C. Griffin & Co., Ltd.; London.
North, S. H.; "The Petroleum Year Book"; Issued yearly, 1921 to date; St. James Press, Ltd.; London.
Nugey, A.L.; "Oil Refinery Specifications"; 1925; The Chemical Publishing Co.; Easton, Pa.; Williams and Norgate; London.

Offermann, H.; "Das norddeutsche Rohölvorkommen in chemisch-physikalisch geologischer Hinsicht"; 1917; Verlag von Friedrich Vieweg & Sohn; Braunschweig.
Ott, Adolph; "Das Petroleum, seine Entdeckung und Verwerthung in den Vereinigten Staaten"; 1875; Verlags-Magazin; Zürich.

Paine, P. M. and *Stroud, B. K.;* "Oil Production Methods"; 1914; Western Engineering Publishing Co.; San Francisco.
Passagez, Albert; "L'azote et le pétrole"; 1926; Gaston Doin; Paris.
Pawlowski, A.; "Les recherches de pétrole en France"; ———; Ch. Béranger; Paris.
Peckham, S. F.; "Production, Technology and Uses of Petroleum and its Products"; 1884; U. S. Government Printing Office; Washington, D. C.

Perutz, H.; "Die Industrie der Mineralöle, des Petroleums, Paraffins, und der Harze"; 1867; Carl Gerold's Sohn; Vienna.

Perutz, H.; "Die Industrie der Mineralöle des Petroleums, Paraffins und Ceresins, nebst den neuesten Fabrikationsmethoden"; 1880; Carl Gerold's Sohn; Vienna.

Petit, Victor; "Guide du sondeur au pétrole"; 2nd Edition, 1922; Ch. Béranger; Paris.

Petroleum Standards Committee; "Standard Methods of Testing Petroleum and Its Products"; 3rd Edition, 1935; W. Speaight & Sons, Ltd.; London.

Petroleum World Publishing Co.; "Petroleum Yearbook 1922"; 1922; Petroleum World Publishing Co.; Los Angeles.

Petrow, G. S. and *Rabinowitsch, A. J.;* "Erdölsulfosäuren und ihre technische Verwendung"; 2nd Edition, 1932; Goschimteschisdal; Leningrad.

Pfeiffer, Karl; "Ueber Erdöl, Bitumen, Oelschiefer und künstliche Schmieröle"; 1922; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; S. Hirzel Verlag; Leipzig.

Pflücke, Maximilian; "Chemisch-Technische Entwicklung auf dem Gebiete der Kohlenwasserstofföle 1928-1932"; 1934; Verlag Chemie, G.m.b.H.; Berlin.

Phelps, R. W. and *Lake, F. W.;* "Petroleum Engineering"; 1st Edition, 1924; Gulf Publishing Co.; Houston, Texas.

Pogue, J. E.; "The Economics of Petroleum"; 1922; John Wiley & Sons, Inc.; New York.

Porter, H. P.; "Petroleum Dictionary"; 1931; The Gulf Publishing Co.; Houston, Texas.

Prudhomme, E. A.; "Le pétrole-roi: sa fabrication industrielle" (Vol. I); 1928; H. Dunod et Cie.; Paris.

Rakusin, M. A.; "Die Untersuchung des Erdöls und seine Produkte"; 1906; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Rakusin, M. A.; "Die Polarimetrie der Erdöl"; 1910; Verlag für Fachliteratur, G.m.b.H.; Berlin.

Redpath, L. V.; "Petroleum in California"; 1900; L. V. Redpath; Los Angeles, Cal.

Redwood, Boverton; "Petroleum: its Production and Use"; 1887; D. Van Nostrand Co. Inc.; New York.

Redwood, Boverton; "Petroleum: a Treatise on the Geographical Distribution and Geological Occurrence of Petroleum and Natural Gas"; 1st Edition (2 Vols.), 1986; 2nd Edition (2 Vols.), 1906; 3rd Edition (3 Vols), 1913; 4th Edition (3 Vols.), 1922; 5th Edition (3 Vols.), 1926; Charles Griffin & Co., Ltd.; London.

Redwood, Boverton and *Eastlake, A. W.;* "Petroleum Technologist's Pocketbook"; 1st Edition, 1915; 2nd Edition, 1924; Charles Griffin & Co., Ltd.; London.

Redwood, Boverton and *Singer, L.;* "Die Mineralöle und ihre Nebenprodukte"; 1898; Verlag von E. Baldamus & Maraun; Leipzig.

Redwood, Iltyd I.; "A Practical Treatise on Mineral Oils and their By-Products"; 1st Edition, 1897; 2nd Edition, 1915; E. & F. N. Spon, Ltd.; London.

Rey, Alphonse; "L'huile de pétrole; connaissance de l'huile de pétrole dans les temps anciens, importance de son exploitation, procédés employés pour

l'extraire et la raffiner, applications diverses de ses dérivés"; 1865; Noblet et Baudry; Paris.

Riche, A. and *Halphen, G.;* "Le Pétrole"; 1896; J. B. Baillière et Fils; Paris.

Ross, Victor; "Petroleum in Canada"; 1917; Published by the Author; Toronto.

Ross, Victor; "Petroleum as a World Power"; 1920; Doubleday, Page & Co.; Garden City, N. Y.

Ross, Victor; "The Evolution of the Oil Industry"; 1920; Published by the Author; New York.

Sachanen, A. N. and *Tilicheyev, M. D.;* "Chemistry and Technology of Cracking" (Translated from the Russian by A. A. Boehtlingk, D. F. Brown and K. T. Steik); 1932; The Chemical Catalog Co., Inc.; New York.

Schieffer, E.; "Bericht über das Naptha-führende Terrain West-Galiziens"; 1865; ———; Vienna.

Schmidt, Ed.; "Das Erdöl Galiziens, dessen Vorkommen und Gewinnung, nebst Beiträgen zur fabrikmässigen Darstellung seiner Produkte"; 1865; Verlag der Gründungs-Comités der Hamburg-Galizischen Petroleum A.-G.; Vienna.

Schneiders, Gottfried; "Die Gewinnung von Erdöl, mit besonderer Berücksichtigung der bergmännischen Gewinnung"; 1928; Verlag von Julius Springer; Berlin.

Schwarz, Robert; "Die Mineralölindustrie Oesterreich-Ungarns"; 1920; Verlag für Fachliteratur, G.m.b.H.; Vienna and Berlin.

Schwarz, Robert; "Petroleum Vademecum" (International Petroleum Tables); 1st Edition, 1922; 2nd Edition, 1923; 3rd Edition, 1926; 4th Edition, 1927; 5th Edition, 1928; 6th Edition, 1929; 7th Edition, 1930; 8th Edition, 1931; 9th Edition (Vols. I and II), 1932; 10th Edition (Vols. I and II), 1933; 11th Edition (Vols. I and II), 1936; Verlag für Fachliteratur, G.m.b.H.; Berlin and Vienna.

Sedlazek, Erwin; "Die Krackverfahren unter Anwendung von Druck (Druckwärmespaltung)"; 1929; Verlag von Julius Springer; Berlin.

Severin, E.; "Das Erdöl, seine Physik, Chemie, Geologie, Technologie und wirtschaftliche Bedeutung"; 1931; ———; Bukarest.

Singer, L.; "Beiträge zur Theorie der Petroleum-Bildung"; 1892; ———; Zurich.

Singer, L.; "Die Technologie des Erdöls und seiner Produkte" (Vol. III); 1911; S. Hirzel Verlag; Leipzig.

Skinner, W. R.; "Oil and Petroleum Manual for 1910"; 1st Issue, 1910; Published yearly thereafter, inclusive of 13th Issue, 1922; "Oil and Petroleum Year Book 1929"; Published 1929; Published yearly thereafter; Same for 1936; Published 1936; W. R. Skinner; 15 Dowgate Hill, Cannon Street, London.

Soulié, Émile and *Haudouin, Hippolyts;* "Le pétrole; ses gisements, ses exploitations, son traitment industriel, ses produits dérivés, ses applications a l' éclairage, et au chauffage"; 1865; Eugene Lacroix; Paris.

Southcombe, J. E.; "Chemistry of the Oil Industries"; 1st Edition, 1913; 2nd Edition, 1926; Constable & Co.; London; D. Van Nostrand Co. Inc.; New York.

Spielmann, P. E.; "Genesis of Petroleum"; 1923; Benn Brothers, Ltd.; London.

Stadnikov, Georg; "Die Entstehung von Kohle und Erdöl"; 1931; Verlag von Ferdinand Enke; Stuttgart.

Steinbrecher, Hans; Stutzer, Otto; and *Moos, August;* "Deutsches Erdöl"; 1934; Verlag von Ferdinand Enke; Stuttgart.

Stigand, I. A.; "Geology of Petroleum; Outlines of the Occurrences"; 1925; Charles Griffin & Co., Ltd.; London.

Strippelmann, Leo; "Die Petroleum-Industrie Oesterreich-Deutschlands"; 1878-9; Verlag von G. Knapp; Leipzig.

Stützer, Otto; "Erdölmuttersubstanz"; 1935; Verlag von Ferdinand Enke; Stuttgart.

Suman, John R.; "Petroleum Production Methods"; 1st Edition, 1921; 2nd Edition, 1922; Gulf Publishing Co.; Houston, Texas.

Sur, F. J. S.; "Oil Prospecting Drilling and Extraction"; 1914; ———; Calgary, Canada.

Sykes, C. P.; "Petroleum in Colorado Territory"; 1865; W. H. Arthur; New York.

Tagliabue, C. J.; "Manual for Inspectors of Petroleum"; 15th Edition, 1920; Newly revised edition (edited by R. M. Wilhelm), 1929; C. J. Tagliabue Mfg. Co.; New York.

Tarbell, Ida M.; "History of the Standard Oil Company" (Vols. I and II); 1925; Macmillan & Co., New York.

Tassart, L. C.; "Exploitation du pétrole"; 1908; H. Dunod et E. Pinat; Paris.

Tate, A. N.; "Petroleum and its Products: An Account of the History, Origin, Composition, Properties, Uses and Commercial Value of Petroleum, etc."; 1863; Davies; London and Liverpool.

Taylor, H. S.; "The A, B, C's of Science in Oil Recovery"; 1927; American Petroleum Institute; New York.

Technicus; "Oilmen's Sundries and How to Make Them"; 1923; Scott, Greenwood & Son; London.

Thomas, H. G.; "La régime du pétrole en France"; 1935; Librairie du recueil Sirey; Paris.

Thompson, A. B.; "Oil Fields of Russia and the Russian Petroleum Industry"; 1908; Crosby, Lockwood & Son; London.

Thompson, A. B.; "Oil Field Developments and Petroleum Mining"; 1st Edition, 1910; 2nd Edition, 1916; Crosby, Lockwood & Son; London.

Thompson, A. B.; "Oil-Field Exploration and Development" (Vols. I and II); 1925; Crosby, Lockwood & Son; London.

Thomson, J. H. and *Redwood, Boverton;* "Handbook on Petroleum"; 1st Edition, 1901; 2nd Edition, 1906; 3rd Edition, 1914; 4th Edition, 1923; Charles Griffin & Co., Ltd.; London.

Thoran, A.; "Die Erdölversorgung unter dem Einflusz der geographischen Verhältnisse der Förderungsländer"; 1932; B. G. Teubner; Leipzig.

Tinkler, C. K. and *Challenger, F.;* "The Chemistry of Petroleum and its Substitutes"; 1st Edition, 1915; 2nd Edition, 1919; D. Appleton & Co.; New York; Crosby, Lockwood & Son; London.

Uren, L. C., "Textbook of Petroleum Production Engineering"; 1st Edition, 1924; "Petroleum Production Engineering—Oil Field Development"; 2nd Edition, 1934; McGraw-Hill Book Co., Inc.; New York.

Van Tuyl, F. M.; "Elements of Petroleum Geology"; 1924; Petroleum Publishing Co.; New York.

Verein deutscher Chemiker; "Von den Kohlen und den Mineralölen. Ein Jahrbuch für Chemie und Technik der Brennstoffe und Mineralöle" (Vol. IV); 1932; Verlag Chemie, G.m.b.H.; Berlin.

Ver Wiebe, W. A.; "Oil Fields in the United States"; 1930; McGraw-Hill Book Co., Inc.; New York.

Vieth, Alexander; "Das Erdöl und seine Verarbeitung"; 1892; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Vlès, F.; "Cours sur la physico-chimie des pétroles"; 1928; Vigot Frères; Paris.

Vlugter, J. C.; "Over de chemische samenstelling von hoogmoleculaire minerale oliën"; 1932; Dissertation; Delft.

Walker, G. T.; "Petroleum, its History, Occurrence, Production, Uses and Tests"; 1915; Imperial Press; Minneapolis, Minn.

Warren, John; "Petroleum and Natural Gas in West Virginia"; 1920; Philips & Van Brunt; New York.

Waterman, H. I.; "Het destilleeren van aardolie"; 1921; Noorduyn & Zoon; Gorinchem, Holland.

Wenger, Léon; "Le petrol; production, industrie, commerce"; 1913; A. Rousseau; Paris.

Westcott, J. H.; "Oil: Its Conservation and Waste"; 4th Edition, 1931; Beacon Publishing Co.; New York.

Williamson, J. W.; "In a Persian Oil Field; A Study in Scientific and Industrial Development; 1928; Ernest Benn, Ltd.; London.

Wischin, R. A.; "Die Naphtene (zyklische Polimethylene des Erdöls)"; 1901; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Witkamp, H.; "De Petroleum"; 1917; Tjeenk Willink & Zoon; Haarlem.

Wright, William; "The Oil Region of Pennsylvania"; 1865; Harper Brothers; New York.

Yancey, J. C.; "Why and Where Oil is Found"; 1920; J. H. Eggers Co.; New York.

(G) DISPERSIONS AND COLLOIDAL CHEMISTRY

Aladin, Dr.; "Technisch Verwendbare Emulsionen"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Alexander, Jerome, "Colloid Chemistry"; Vol. I, 1926; Vol. II, 1928; Vol. III, 1931 (Section entitled "Asphalt", by *F. J. Nellensteyn,* p. 535); Chemical Catalog Co., Inc.; New York.

Auskunft- und Beratungstelle für Teerstrassenbau e.V.; "Kaltteer"; 1936; Auskunft- und Beratungstelle für Teerstrassenbau e.V.; Essen, Germany.

Clayton, William; "Theory of Emulsions and their Technical Treatment"; 1st Edition, 1922; 2nd Edition, 1927; 2nd Edition, Revised, 1928; 3rd Edition, 1935; J. & A. Churchill, Ltd.; London.

Garner, F. H., Gabriel, L. G. and *Prentice, H. J.;* "Modern Road Emulsions"; 1933; The Carriers' Publishing Co., Ltd.; London.

Kuhn, Alfred; "Wörterbuch der Kolloidchemie"; 1932; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Lange, Otto; "Technik der Emulsionen"; 1929; Verlag von Julius Springer; Berlin.

Liesegang, R. E., "Kolloid-chemischen Technologie"; 1st Edition, 1927; 2nd Edition, 1932 (Section entitled "Asphalte und Teere", by *Albrecht von Skopnik,* p. 616); Verlag von Theodor Steinkopff; Dresden and Leipzig.

Price, T. M.; "The Preparation of Emulsions of Crude Petroleum"; 1905; U. S. Animal Industry Bureau Circular No. 89; U. S. Dept. Agriculture, Government Printing Office; Washington, D. C.

Reichverbandes der deutschen Kaltasphaltunternehmungen e.V.; "Kaltasphalt in seinen verschiedenen Anwendungsgebieten"; 1934; Druckschrift des Reichsverbandes der deutschen Kaltasphaltunternehmungen e.V.; Berlin.

Scheuermann, Magistratsbaurat; "Aus der Praxis der Kaltasphaltierungen"; 1st Edition, 1933; 2nd Edition, 1934; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Scheuermann, Magistratsbaurat and *Knollenberg, R.;* "Zur Frage der Kaltasphaltierungen"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Symposium; "Technical Aspects of Emulsions"; 1935; A. Harvey; London.

Travis, P. M.; "Mechano-Chemistry and the Colloid Mill"; 1928; Chemical Catalog Co., Inc.; New York.

Weber, Hans and *Bechler, Hermann;* "Ueber den Zerfall der bituminösen Strassenbau-Emulsionen durch Berührung bit Gestein"; 1932; Sonderdruck aus "Asphalt- und Teer-Strassenbautechnik", Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Wilkinson, F. and *Forty, F. J.;* "Bituminous Emulsions for Use in Road Works"; 1932; Contractors' Record, Ltd.; London.

Zsigmondy, Richard; "Kolloidchemie"; 5th Edition, 1925; Verlag von Otto Spamer; Leipzig.

(H) PAVING MATERIALS

Abbott, J. W.; "Use of Mineral Oil in Road Improvement"; 1903; U. S. Dept. of Agriculture, U. S. Government Printing Office; Washington, D. C.

Abbott, N. B.; "Bituminous Concrete Pavements as Laid in the United States"; 2nd Edition, 1876; R. M. Whiting & Co.; Brooklyn, N. Y.

Agg, T. R.; "Construction of Roads and Pavements"; 1st Edition, 1916; 2nd Edition, 1920; 3rd Edition, 1924; 4th Edition, 1929; McGraw-Hill Book Co., Inc.; New York.

Aitken, Thomas; "Road Making and Maintenance"; 1st Edition, 1900; 2nd Edition, 1908; Charles Griffin & Co., Ltd.; London.

Allen, A. T.; "Footpaths; Their Maintenance, Construction and Cost"; 1st Edition, 1900; 2nd Edition, 1906; Sanitary Publishing Co. Ltd.; London.

Allen, A. T.; "New Streets: Laying Out and Making Up"; 1904; Sanitary Publishing Co. Ltd.; London.

American Association of State Highway Officials; "Tentative Standard Specifications for Highway Materials and Methods of Sampling and Testing"; 1932; The American Association of State Highway Officials; Washington, D. C.

American Wood-Preservers' Association; "Handbook on Wood Preservation"; 1916; American Wood-Preservers' Association; Baltimore, Md.

Andes, L. E.; "Die Beseitigung des Staubes"; 1908; A. Hartleben's Verlag; Vienna and Leipzig.

Arbeitsgemeinschaft der Bitumen-Industrie e.V.; "Strassenbau und Bitumen"; 1934; Arbeitsgemeinschaft der Bitumen-Industrie e.V.; Berlin.

Asphalt Association and *Graefe, Edmund;* "Asphalt; kleines Taschenbuch für den praktischen Strassenbauer"; 4th Edition, 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Audouin, Paul; "(I) Mortier de goudron; (II) Quelques mots à propos du goudronnage des routes" (Extraits du "Cosmos", Janvier et Avril 1906); 1906; Impr. de P. Feron-Vrau; Paris.

Baker, Ira O.; "Treatise on Roads and Pavements"; 1st Edition, 1903; 1st Edition, Revised, 1908; 2nd Edition, 1913; 2nd Edition, Revised, 1914; 3rd Edition, 1918; 4th Edition, 1922; D. Van Nostrand Co. Inc.; New York; Chapman & Hall, Ltd.; London.

Ballen, Dorothy; "Bibliography of Road-Making and Roads in the United Kingdom" (Including a bibliography compiled by Mr. and Mrs. Sidney Webb); 1914; P. S. King & Son; London.

Bartel, A.; "Taschenbuch für den Gesamten Strassen- und Wegebau"; Published Yearly; 1st Edition, 1910; 23rd Edition, 1932; Bock & Co.; Berlin.

Bartholomäi, Dr.-Ing.; "Kompression und Kornverfeinerung im Teer- und Asphaltstrassenbau" (Vol. 12, Technischen Hochschule zu Stuttgart); 1935; Verlag Konrad Triltsch; Würzburg.

Barton, W. H. and *Doane, L. H.;* "Sampling and Testing of Highway Materials"; 1st Edition, 1925; McGraw-Hill Book Co., Inc.; New York.

Bateman, J. H.; "Highway Engineering"; 1st Edition, 1928; 2nd Edition, Revised, 1934; Chapman & Hall, Ltd.; London; John Wiley & Sons, Inc.; New York.

Bauer, Edward E.; "Highway Materials"; 1st Edition, 1928; 2nd Edition, 1932; McGraw-Hill Book Co., Inc.; New York.

Baumeister, Reinhard; "Städisches Strassenwesen und Städtreinigung"; 1890; Ernest Toeche Publishing Co.; Berlin.

Bayerische oberste Bauberhörde; "Die bayerischen Staatstrassen"; 1925; Selbstverlag; Munich.

Beckwith, Arthur; "Report on Asphalt and Bitumen, as Applied to the Construction of Streets and Sidewalks in Paris"; 1868; U. S. Government Printing Office; Washington, D. C.

Bennett, J. M.; "Roadside Development"; 1929; Macmillan & Co.; New York.

Besson, F. S.; "City Pavements"; 1st Edition, 1923; McGraw-Hill Book Co., Inc.; New York.

Betz, Louis; "Automobil und Fahrstrasse in ihre Wechselwirkungen"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Bierhalter, Willi; Krüger, Karl; Ohse, Ernst; von Skopnik, Albrecht; and *Stöcke, Kurt;* "Wie prüft man Strassenbaustoffe?"; 1932; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Birk, Alfred, "Der Wegebau"; Vol. I: "Erdbau und Strassenbau", 1904; Vol. II: Eisenbahnbau", 1906; Vol. III: "Der Tunnelbau", 1911; Vol. IV: "Linienführung der Strassen und Eisenbahnen", 1915; Vol. V: "Bauleitung", 1922; Vol. VI: "Signal- und Sicherungsanlagen bei Eisenbahnen", 1924; Franz Deuticke; Leipzig and Vienna.

Birk, Alfred; "Die Strasse, ihre Verkehrs- und bautechnische Entwicklung im Rahmen der Menschheitsgeschichte"; 1935; Adam Kraft Verlag; Karlsbad-Drahowitz.

Blanchard, A. H.; "American Highway Engineers' Handbook"; 1st Edition, 1919; John Wiley & Sons, Inc.; New York.

Blanchard, A. H.; "Elements of Highway Engineering"; 1st Edition, 1915; *Blanchard, A. H.,* and *Morrison, R. L.;* 2nd Edition, 1928; John Wiley & Sons, Inc.; New York.

Blanchard, A. H. and *Drowne, H. B.;* "Highway Engineering as Presented at the Second International Road Congress, Brussels, 1910"; 1911; John Wiley & Sons, Inc.; New York.

Blanchard, Arthur H. and *Drowne, H. B.;* "Text Book on Highway Engineering"; 1st Edition, 1914; John Wiley & Sons, Inc.; New York.

Bloodgood, S. DeWitt; "A Treatise on Roads; their History, Character and Utility"; 1838; O. Steele; Albany, N. Y.

Bösenberg, H.; "Die Praxis des neuzeitlichen Asphaltstrassenbaus" (Volume I); 1928; Brend'amour, Simhart & Co.; Munich.

Boulnois, H. P.; "The Construction of Carriageways and Footways"; 1895; Biggs & Co.; London.

Boulnois, H. P.; "Practical Road Engineering for the New Traffic Requirements"; 1st Edition, 1910; 2nd Edition, 1921; The St. Bride's Press, Ltd.; London.

Boulnois, H. P.; "A Glossary of Road Terms"; 1914; The St. Bride's Press, Ltd.; London.

Boulnois, H. P.; "Modern Roads"; 1919; Edwin Arnold; London.

Bradley, Harold and *Hancock, C. C.;* "Modern Road Making"; 1928; Contractors' Record, Ltd.; London.

Bredtschneider, K.; "Asphalt und Teer im Dienste des Strassenbaus"; 1919; Carl Heymann's Verlag; Berlin.

Brix, Dr.-Ing. and *Klein, Heinrich;* "Jahrbuch für Strassenbau – 1929"; 1929; "Jahrbuch für Strassenbau – 1930-1"; 1932; "Jahrbuch für Strassenbau – 1932"; 1933; Verlag von Richard Pflaum; Munich.

Burgoyne, Sir J. F.; "Rudiments of the Art of Constructing and Repairing Common Roads"; 1st Edition, 1850; 2nd Edition, 1855; 3rd Edition, 1861-2; J. Weale; London.

Burrows, A. J.; "The Maintenance and Construction of Roads"; 1884; W. Rider & Son; London.

Byrne, A. T.; "Treatise on Highway Construction"; 1st Edition, 1892; 2nd Edition, 1893; 3rd Edition, 1896; 4th Edition, 1900; 5th Edition, 1907; *Byrne, A. T.,* and *Phillips, A. E.;* 5th Edition, Revised, 1908; 6th Edition, 1913; John Wiley & Sons, Inc.; New York.

Byrne, A. T.; "Modern Road Construction"; 1st Edition, 1917; 2nd Edition, 1919; American Technical Society; Chicago.

Chatburn, Geo. R.; "Highway Engineering, Rural Roads and Pavements"; 1921; John Wiley & Sons, Inc.; New York.

Clark, D. K.; "The Construction of Roads and Streets"; 1st Edition, 1877; 2nd Edition, 1881; Crosby, Lockwood & Son; London.

Coane, J. M., Coane, H. E. and *Coane, J. M. Jr.;* "Australasian Roads"; 1st Edition, 1908; 2nd Edition, 1915; George Robertson & Co.; Melbourne, Sydney, Adelaide and Brisbane.

Cordier, Joseph; "Ponts et chaussées" (Vols. I and II); 1923-8; Reboux-Leroy; Lille, France.

Dammann, Karl; "Essener Asphalt"; 1926; ————; Essen (Ruhr).

Dammann, Karl; "Die Kornzusammensetzung im Dammann-asphalt" (Fortschritte im Städte- und Strassenbau, Band 6); 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Deutscher Normenausschuss; "Bitumen und Teer enthaltende Massen für Strassenbau und ähnliche Zwecke"; 2nd Edition, 1936; Beuth-Verlag; Berlin.

Deutscher Strassenbauverband; "Vorschriften des deutschen Strassenbauverbandes für die Beschaffenheit, Probenahme und Untersuchung von bituminösen Bindemitteln im Strassenbau"; 4th Edition, 1932; 5th Edition, March 1933; Verlag der Verkehrstechnik; Berlin.

Dietrich, Emil; "Die Baumaterialien der Asphaltstrassen"; 1881; Verlag von Julius Böhne; Berlin.

Dietrich, Emil; "Die Asphaltstrassen"; 1882; Verlag von Julius Böhne; Berlin.

Dubosque, J.; "Réparation et entretien des chaussées"; 1898; Ch. Béranger; Paris.

Durham, H. W.; "Street Paving and Maintenance in European Cities"; 1914; Press of C. S. Nathan; New York.

Ebberts, A. R. and *Johnstone, Hugh;* "Bituminous Pavements; An Introduction to the Art"; 1936; Published by the Authors; Pittsburgh, Pa.

Ehlgötz, Hermann, and *Krüger, Karl,* "Fortschritte im Städtebau und Strassenbau"; Vol. I, by *Karl Krüger,* "Ueber mineralische Baustoffe", 1929; Vol. II by *Paul Wichert,* "Füller und Bitumen", 1929; Vol. III by *Ernst Johanssen,* "Walzasphalt – Theorie und Praxis", 1930; Vol. IV by *Enno Müller-Essens,* "Grundgedanken zur Rationalisierung des Verkehrswesens im Rhein-Ruhr-Bezirk", 1930; Vol. V by *E. Runge,* "Grundsätzliches zur Problem der Erweiterung Gross – Berlins", 1930; Vol. VI by *Karl Dammann* and *Ing. Lesser,* "Dammann-Asphalt", 1931; Vol. VII by *E. Kuthe,* "Der Aufbau von Makadamstrassen unter Verwendung von Teer und Asphalt", 1931; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Eickner, Gustav; "Wirtschaftlichkeit und Produktivität des Ausbaues Strassen für den Kraftwagenverkehr in der Prov. Hannover"; 1927; Verlag Martin Boerner; Halle (Saale).

Eldridge, M. O.; "Progress of Road Building in the United States"; 1900; U. S. Dept. Agriculture, Government Printing Office, Washington, D. C.

Ellice-Clark, E. B.; "Asphalt and its Applications to Street Paving"; 1879; ———; London.

Ende, Paul Am.; "Der Strassenstaub und seine Bekämpfung"; 1910; C. Richard Gärtnersche Buchdeuckerei (Heinrich Niescher); Dresden.

Exchaquet, H.; "Dictionnaire des ponts et chaussees"; 1787; Mourer et Cie.; Lausanne.

Fachleuten; "Strassenteer und Teerstrassenbau"; 1st Edition, 1928; 2nd Edition, 1930; Auskunfts- und Beratungsstelle für Teerstrassenbau e. V. Essen; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Fachleuten; "Vorschriften des deutschen Strassenbau-Verbandes über die Eigenschaften und Untersuchungen von Bitumen für Strassenbau"; 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Fachleuten; "Vorschriften über die Eigenschaften und Untersuchungen von Strassenbau-Emulsionen des deutschen Strassenbau-Verbandes"; 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Fachleuten; "Teerstrassenbau und Landstrassen" (Vols. 1 to 4 incl.); 1930; Auskunft- und Beratungsstelle fur Teerstrassenbau e. V. Essen; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Fachleuten; "Strassenbau und Bitumen"; 1934; Arbeitsgemeinschaft der Bitumenindustrie e.V.; Berlin.

Fachleuten, "Teerstrassenbau in Einzeldarstellungen" (Vol. 5, Teertränk- und Teerstreumakadam; Vol. 6, Teermischmakadam; Vol. 7, Dammann-Bauweisen; Vol. 8, Teerbeton); 1934; Auskunft- und Beratungstelle für Teerstrassenbau e.V.; Essen (Selbstverlag).

Foote, C. E.; "Practical Road Building"; 1917; D. McKay; Philadelphia.

Forbes, R. J.; "Notes on the History of Ancient Roads and their Construction"; 1934; N. V. Noord-Hollendsche Uitgers-Mij.; Amsterdam, Holland.

Freese, H.; "Die Holzpflaster in London"; 2nd Edition, 1924; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Friedrich, Paul; "Silnični Stroje" (Vol. II of Přiručni Silnični Knihovna, Publication Bureau for Roadbuilding); 1934: Šiwel and Beran; Prague (Praha), Czechoslovakia.

Frost, Harwood; "The Art of Roadmaking"; 1910; Engineering News Publishing Co.; New York.

Funk, P. G.; "Das Kunststrassenwesen"; 1936; Verlag Martin Boerner; Halle (Saale).

Gamann, H.; "Die Unterhaltung der Wege und Fahrstrassen"; 2nd Edition, Revised; 1915; P. Parey; Berlin.

Gautier, Hubert; "Traité de la construction des chemins, où il est parlé de ceux des romains et de ceux des modernes, suivant qu'on les practique en France"; 1750; Cailleau; Paris.

Gavrian, P. Le; "Les chaussées modernes"; 1st Edition, 1922; 2nd Edition, 1935; J. B. Baillière et Fils; Paris.

Geissler, Wilhelm; "Der Strassenbau" (Sammlung Göschen, Vol. 740); 1935; Walter de Gruyter & Co.; Berlin and Leipzig.

Genzmer, Ewald; "Die städtischen Strassen" (Vols. I and II); 1897-1900; A. Bergsträsser; Stuttgart.

Gerlach, Ernst; "Die Prüfung der Mischvorgänge im bituminösen Strassenbau"; 1937; V. D. I. Verlag; Berlin.

Gillespie, W. M.; "Principles and Practice of Road Making"; 1st Edition, 1847; 2nd Edition, 1848; 3rd Edition, 1849; 8th Edition, 1853; 9th Edition, 1860; 10th Edition, 1871; Barnes and Burr; New York.

Gillette, H. P.; "Economics of Road Construction"; 1st Edition, 1901; 2nd Edition, 1906; Engineering News Publishing Co.; New York.

Gillmore, Q. A.; "A Practical Treatise on the Construction of Roads and Pavements"; 4th Edition, 1876; 5th Edition, 1882; 6th Edition, 1888; D. Van Nostrand Co. Inc.; New York.

Goldsmith, H. E.; "Modern Road Construction and Maintenance"; 1st Edition, 1920; 2nd Edition, 1923; Kelly & Walsh, Ltd.; Hongkong.

Goldsmith, H. E.; "Practical Road Engineering"; 1925; Crosby, Lockwood & Son; London.

Goodell, J. M.; "Location, Construction and Maintenance of Roads"; 1918; McGraw-Hill Book Co., Inc.; New York.

Graefe, Edmund; "Die Bedeutung der Laboratoriumsarbeit beim Bau bituminöser Strassen"; 1926; Sonderdruck aus der Asphalt– und Teerindustriezeitung; Berlin.

Graefe, Edmund; "Asphalt – Kleines Taschenbuch für den praktischen Strassenbauer"; 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Green, J. W. and *Ridley, C. N.;* "The Science of Roadmaking"; 1927; Crosby, Lockwood & Son; London.

Greenwell, Allan and *Elsden, J. V.;* "Roads; Their Construction and Maintenance"; 1901; D. Appleton & Co.; New York.

Grengg, Roman; "Ueber zweckmäszige Prüfungsverfahren von Gesteinen für Strassenbauzwecke"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Haller, K.; "Uebersicht über den Stand des amerikanischen Strassenbauwesens"; 1926; Studiengesellschaft für Automobilstrassenbau; Charlottenburg.

Harger, W. G.; "Rural Highway Pavements, Maintenance and Reconstruction"; 1st Edition, 1924; McGraw-Hill Book Co., Inc.; New York.

Harger, W. G. and *Bonney, E. A.;* "Highway Engineers' Handbook"; 1st Edition, 1912; 2nd Edition, 1916; 3rd Edition, 1919; 4th Edition, 1927; McGraw-Hill Book Co., Inc.; New York.

Hasluck, P. N.; "Road and Footpath Construction"; 1904; Cassell & Co.; London.

Heeb, A. and *Kölmel, A.;* "Strassenbau"; 1934; Verlag Teubner, Leipzig.

Heinrici, C. and *Temme, Th.;* "Strassenbau und Bitumen"; 1935; Arbeitsgemeinschaft der Bitumen-Industrie, e.V.; Berlin.

Heinrici, C.; Westmeyer, R.; Geissler, Wilhelm; Riedel, Walter; Temme, Th.; Bösenberg, H.; Bandte, G.; v. Skopnik, Albrecht; Becker, Walter; Ziegs, Carl and *Bierhalter, Willi;* "Bitumen in der Praxis"; 1934; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin – Lichterfelde.

Hentrich, H.; "Reise nach London zum Studium der Automobilstrassen"; 1924; Studiengesellschaft für Automobilstrassenbau; Charlottenburg.

Hentrich, H.; "Reise nach der Schweiz zum Studium des Teerstrassenbaues"; 1925; Verlag von Wilhelm Knapp; Halle (Saale).

Hentrich, H.; "Die neuzeitliche Strassenbau"; Vol. I by *Ing. Rappaport* and *K. Haller,* "Allgemeine Strassenplannung", 1927; Vol. II by *Ing. Knipping,* "Konstruktion und Ausführung", 1930; Vol. III by *Ing. Knipping,* "Steinstrassen", 1928; Vol. IV by *Ing. Kleinlogel,* "Betonstrassen", 1929; Vol. V by *H. Hentrich,* "Teerstrassen", 1927; Vol. V (2nd Edition) by *H. Hentrich* and *Baurat Grossjohann,* "Teerstrassen", 1934; Vol. VI by *Emil Ilse,* "Asphaltstrassen", 1930; Vol. VII by *Dr. Heymann,* "Verwaltung und Wirtschaft", 1929; Verlag von Wilhelm Knapp; Halle (Saale).

Herrmann, Paul and *Bierhalter, Willi;* "Tätigkeitsbericht für das Geschäftsjahr 1931"; 1933; "Tätigkeitsbericht für das Geschäftsjahr 1932"; 1934; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin – Lichterfelde.

Herschel, Clemens and *North, E. P.;* "Science of Road-Making"; 1894; Engineering News Publishing Co.; New York.

Hlauschek, Hans; "Naphthen- und Methanöle, ihre geologische Verbreitung und Enstehung"; 1937; Verlag von Ferdinand Enke; Stuttgart.

Höpfner, Wilhelm; "Bericht über die Besichtigung von Teerstrassen"; 1926; Studiengesellschaft für Automobilstrassenbau; Charlottenburg.

Hotz, Edgar; "Untersuchungen über die Wirkungsweise des Steinkohlenteers als bituminöses Bindemittel bei Teermineraldecken"; 1928; Friedrich Gutsch; Karlsruhe.

Hubbard, Prévost; "Dust Preventatives and Road Binders"; 1910; John Wiley & Son, Inc.; New York.

Hubbard, Prévost; "Laboratory Manual of Bituminous Materials"; 1916; John Wiley & Sons, Inc.; New York.

Hubbard, Prévost; "Highway Inspectors' Handbook"; 1919; John Wiley & Sons, Inc.; New York.

Hubbard, Prévost and *Reeve, C. S.;* "Methods for the Examination of Bituminous Road Materials" (Bulletin No. 314); 1915; U. S. Dept. of Agriculture, Government Printing Office, Washington, D. C.

Hubert, Octave; "Traité d'asphaltage"; 1932; Ch. Béranger (Librairie Polytechnique); Paris and Liege.

Hughes, Thomas; "The Practice of Making and Repairing Roads"; 1838; John Weale Architectural Library; London.

Jentsch, Stadtbaurat; "Aussichten und Aufgaben für den deutschen Strassenbau"; 1925; Studiengesellschaft für Automobilstrassenbau; Charlottenburg.

Johannesson, Sigvald; "Highway Economics"; 1st Edition, 1931; McGraw-Hill Book Co., Inc.; New York.

Johannson, Ernst; "Walzasphalt, Theorie und Praxis"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Johannson, Ernst; "Bituminöse Dauerdecken" (Vol. 2 of "Städtebau und Strassenbau"); 193; Carl Heymann's Verlag; Berlin.

Judson, W. P.; "City Roads and Pavements for Oswego, New York"; 1st Edition, 1894; "City Roads and Pavements Suited to Cities of Moderate Size"; 2nd Edition, 1902; 3rd Edition, 1907; 4th Edition, 1909; Engineering News Publishing Co.; New York.

Judson, W. P.; "Road Preservation and Dust Prevention"; 1908; Engineering News Publishing Co.; New York.

Kerkhof, B. J.; "Asphalt und Teerstrassen", 1st Edition, 1925; 2nd Edition, 1927;

Kerkhof, B. J., and *Ilse, Emil;* 3rd Edition, 1929; Verlag von Julius Springer; Berlin.

Kerkhof, B. J.; "Wegenbouw"; 1931; Kosmos; Amsterdam.

Klein, Heinz; "Strassenteerungen"; 1934; Bayerische Druckerei und Verlagstanstalt, G.m.b.H.; München.

Klinkmann, G. H.; "Beitrag zur Kenntnis der Teer-Erdölbitumen-Mischungen für den Strassenbau"; 1931; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Klinner, G.; "Strassen- und Wegebau" (Vol. III); 1927; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Klose, Georg; "Asphalt und Asphaltmaschinen im Strassenbau"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Knight, B. H.; "Road Aggregates; Their Uses and Testing" (Vol. III of The Roadmakers' Library); 1934; Edward Arnold & Co.; London.

Knipping, F.; Gölz, K. and *Mittmeyer, H.;* "Der Strassenbau der Vereinigten Staaten von Amerika"; 1934; Verlagsbuchhandlung Julius Springer; Berlin.

Kohler, Hellmut; "Teerfeinmineraldecken" (Städtebau und Strassenbau, Vol. IV); 1934; Technischen Hochschule Berlin; Berlin.

Kröcker, Heinrich; "Der Teerstrassenbau"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Krüger, Karl; "Asphaltstrassenbau"; 1926; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Krynine, D. P.; "Text-Book on Highway Engineering"; 1st Edition, 1926; 2nd Edition, 1929; Published by the Author; Moscow.

Kühn, I.; "Beitrag zur Frage der weiteren Ausbildung der Maschinenarbeit beim Strassenbau"; 1928; Verlag von Martin Boerner; Halle (Saale).

Kummer, F. A.; "Creo-Resinate Wood Pavements for Streets and Bridges"; 1903; United States Wood Preserving Co.; New York.

Kuthe, Erich; "Der Aufbau von Makadamstrassen, unter Verwendung von Teer und Asphalt"; 1931; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

von Laissle, Friedrich; "Der Strassenbau Einschliesslich der Strassenbahnen" (Handbuch der Ingenieurwissenschaften, Vol. I); 2nd Edition, 1898; 3rd Edition, 1902; 4th Edition, 1912; Verlag von Wilhelm Engelmann; Leipzig.

Langsdorf, K. C.; "Anleitung zum Strassen- und Brückenbau"; 1817; Schwann- und Göszischen Buchhandlung; Mannheim and Heidelberg.

Larrañaga, P. J. M.; "Successful Asphalt Paving"; 1926; Richard Clay & Sons, Ltd.; London.

Latham, Frank; "Construction of Roads, Paths, and Sea Defences"; 1903; The Sanitary Publishing Co., Ltd.; London.

Law, Henry; "Rudiments of the Art of Constructing and Repairing Common Roads"; 1st Edition, 1850; 2nd Edition, 1855; John Weale Architectural Library; London.

Law, Henry and *Clark, D. K.;* "Construction of Roads and Streets"; 1st Edition, 1877; 2nd Edition, 1881; Lockwood & Son; London.

Leeming, E. L.; "Road Engineering"; 1924; A. Constable & Co., Ltd.; London.

Lefebvre, Georges; "Voie publique"; 1896; C. Dunod & P. Vicq.; Paris.

Lendway, A. B.; "Dérivés du pétrole fossile roumains"; 1900; E. Göbl; Bucarest.

Letouzé P. and Loyeau, P.; "Traité practique des travaux en asphalte"; 1897; F. Bernhard et Cie.; Paris.

Levsen, Paul and *Rentsch, Bernhard;* "Der angemessene Preis im Strassenbau"; 1936; Otto Elsner Verlagsgesellschaft; Berlin.

Liebermann, C.; "Der Landstrassenbau"; 2nd Edition, 1920; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Loewe, F.; "Die Bekämpfung des Strassenstaubes"; 1910; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Loewe, Ferdinand; "Strassenbaukunde" (Handbuch der Ingenieurwissenschaften); 1st Edition, 1906; 2nd Edition, 1913; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Love, E. G.; "Pavements and Roads; their Construction and Maintenance"; 1890; Reprinted from the Engineering & Building Record; New York.

Lüer, Hans; "Die Teerstrassenbau-Tagung in Godesberg (vom 15 Oktober 1927)"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Lüer, Hans; "Beiträge zur Teerstrassenbauforschung, insonderheit Studien über die Mischung von Teer und Asphalt"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Lüer, Hans; "Der Teerbeton der Gesellschaft für Teerstrassenbau"; 1930; Verlag Boeckling & Müller; Essen.

Lüer, Hans; "Teerstrassenbau unter besonderer Berücksichtigung der Hochofenschlacke"; 1931; Allegemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Lüer, Hans; "Stampfbare Teerfeinkornbeläge"; 1934; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Lüer, Hans; "Teerstrassen" (Vorträge aus den Ausschuszsitzungen des Ausschusses Teerstrassen); 1934; Verlag Stufa; Berlin (Charlottenburg).

Macadam, J. L.; "Remarks on the Present System of Road-Making with Observations Deduced From Practice and Experience"; 1st Edition, 1821; 2nd Edition, 1823; 3rd Edition, 1827; Longmans & Co.; London.

Machinek, Paul; "Neuzeitlicher Asphaltstrassenbau in Groszberlin"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Malo, Léon; "Note sur les chaussées en asphalte comprimé"; 1864; Eugène Lacroix; Paris.

Malo, Léon; "Guide practique pour la fabrication et l'application de l'asphalte et des bitumes"; 1st Edition, Paris, 1861; 2nd Edition, 1866; Eugène Lacroix; Paris.

Malo, Léon; "On Asphalte Roadways"; 1886; E. F. & N. Spon, Ltd.; London.

Maxwell, W. H.; "The Construction of Roads and Streets"; 1899; A. Constable & Co., Ltd.; London.

Mayn, L.; "Der Asphalt und seine Bedeutung für den Strassenbau groszer Städte"; 1872; Buchhandlung des Waisenhauses; Halle (Saale).

Merrill, F. J. H.; "Road Materials and Road Building in New York"; 1897; University of the State of New York; Albany, N. Y.

Milke, H.; "Der Strassenbau" (Vol. 24 of "Musterbetriebe deutscher Wirtschaft"; Die Strassenbau A.-G.); 1934; Verlag J. J. Arndt; Leipzig.

Miozzi, Eugenio; "Manufatti Stradli"; 1929; Ulrico Hoepli; Milano.

Morrison, C. E.; "Highway Engineering"; 1st Edition, 1908; John Wiley & Sons, Inc.; New York.

Müller, A.; "Taschenbuch für den gesamten Strassen- und Wegebau"; 1st Edition, 1931; 2nd Edition, 1934; Verlag Block & Co., G.m.b.H.; Berlin.

Nellensteyn, F. J. and *Loman, R.;* "Asfaltbitumen en Teer. Theorie en Practijk der bitumineuze Wegdekken"; 1932; D. B. Centen; Amsterdam.

Neumann, E., "Kritische Betrachtungen über den gegenwärtigen Stand des Strassenbauwesens in den Vereinigten Staaten von Nordamerika"; 1926; Verlag von Wilhelm Ernst & Sohn; Berlin.

Neumann, E.; "Der neuzeitlicher Strassenbau"; 1st Edition, 1927; 2nd Edition, 1932; Verlag von Julius Springer; Berlin.

Neumann, E. and *Feuchtinger;* "Bericht über eine Studienreise in den Vereinigten Staaten von Nordamerika"; 1926; Studiengesellschaft für Automobilstrassenbau; Charlottenburg.

Niedner, F.; "Die Strassenreinigung in den deutschen Städten"; 1911; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

North, Edw. P.; "Construction and Maintenance of Roads"; 1894; Engineering News Publishing Co.; New York.

Oberbach, J.; "Asphalt- und Teerstrassendecken, ihre Fundamentierung und Zusammensetzung"; 1st Edition, 1930; 2nd Edition, 1931; Verlag von Martin Boerner; Halle (Saale).

Osthoff, Georg; "Der Strassen- und Wegebau"; 1882; Spohr, Gebr.; Leipzig.

Page, L. W.; "Roads, Paths and Bridges"; 1912; Sturgis & Walton Co.; New York.

Parnell, H. B.; "A Treatise on Roads"; 1st Edition, 1833; 2nd Edition, 1838; J. Ridgway; London.

Peckham, S. F. and *Klein, O. H.;* "Asphalt Paving"; 1904; Report of the Commissioners of Accounts; New York.

Penfold, Charles; "A Practical Treatise on the Best Modes of Making Roads" ("British Husbandry"); 1834; John F. Burke; London.

Piepmeier, B. H.; "Surface Oiling of Earth Roads"; 1915; Schnepp & Barnes; Springfield, Ill.

Pöpel, F.; "Die moderne Strassenbau"; 1929; Verlag von Martin Boerner; Verlag von Wilhelm Knapp; Halle (Saale).

Presser, Siegfried; "Die Fahrbahnbefestigung der Reichs- und Landstrassen in Wort und Bild"; 1934; Union Deutsche Verlagsgesellschaft; Berlin.

Radford, Thomas; "The Construction of Roads and Pavements"; 3rd Edition, 1924; 4th Edition, 1929; McGraw-Hill Book Co., Inc.; New York.

Reiner, Wilhelm; "Handbuch der neuen Strassenbauweisen mit Bitumen, Teer und Portland-Zement als Bindemittel"; 1929; Verlag von Julius Springer; Berlin.

Richardson, Clifford; "The Modern Asphalt Pavement"; 1st Edition, 1905; 2nd Edition, 1908; John Wiley & Sons, Inc.; New York.

Richardson, Clifford; "Bituminous Materials for Use in and on Road Surfaces, and Means of Determining their Character"; 1909; New York Testing Laboratory; New York.

Richardson, Clifford; "Asphalt Construction for Pavements and Highways"; 1913; Mc-Graw Hill Book Co., Inc.; New York.

Richardson, Clifford and *Forrest, C. N.;* "Methods for the Examination of Bituminous Materials for Road Construction"; 1909; New York Testing Laboratory; New York.

Rockwell, A. P.; "Roads and Pavements in France"; 1st Edition, 1896; John Wiley & Sons, Inc.; New York.

Roux, Onésime; "Routes et chemins vicinaux"; 1901; H. Dunod et Cie; Paris.

Ryves, R. A.; "The King's Highway; the Nature, Purpose and Development of Roads and Road Systems"; 1908; The St. Bride's Press, Ltd.; London.

Salkield, Tom; "Roadmaking and Road Using"; 1927; Sir Isaac Pitman & Sons, Ltd.; London.

Schaar, W.; "Die Beanspruchung der Strassen durch die Kraftfahrzeuge"; 1925; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; Zementverlag; Charlottenburg.

Schenck, Rudolf; "Die Kraftwagenstrasse, ein Leitfaden für den modernen Strassenbau"; 1925; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Schenck, Rudolf; "Fahrbahnreibung und Schlüpfrigkeit der Strassen im Kraftwagenverkehr"; 1930; M. Krayn; Berlin.

Schenck, Rudolf; "Die Prüfung von Strassenbaustoffen und neueren Strassendecken"; 1932; Verlag von Wilhelm Knapp; Halle (Saale).

Scheuermann, Magistratsbaurat, "Zur Frage der Kaltasphaltierungen"; 1st Edition, 1926; *Scheuermann, Magistratsbaurat,* and *Knollenberg, R.;* 2nd Edition, 1928; Allgemeiner Industrie-Verlag G.m.b.H.; Berlin.

Scheuermann, Magistratsbaurat; "Schutz auch dem deutschen Hartasphalt"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Scheuermann, Magistratsbaurat; "Steingeschläg und Strassenteer"; 1930; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Scheuermann, Magistratsbaurat; "Aus der Praxis der Kaltasphaltierungen"; 1933; Allgemeiner Industrie Verlag, G.m.b.H.; Berlin.

Schewior, George; "Handbuch der Strassen- und Wegeführungen auf dem Lande"; 1913; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Schmid, Carl; "Asphalt, Teer, Oel im Strassenbau"; 1905; Konrad Wittwer Verlag; Stuttgart.

Schmidt, Hugo and *Herrmann, Paul;* "Die Prüfung von Stampfasphalt und anderen Strassendecken mit bituminösen Bindemitteln"; 1915; Carl Heymann's Verlag; Berlin.

Schmitt, Johann; "Die Bedeutung von Bindemittel und Mineral im bituminösen Strassenbau"; 1936; Triltsch & Huther; Berlin.

Schneider, Eduard; "Moderner Strassenbau"; 1st Edition, 1926; 2nd Edition, 1928; 3rd Edition, 1933; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Schubarth, E. O.; "Ueber Asphaltstrassen"; 1881; Polytechn. Buchhandlung; Berlin.

Schubarth, E. O.; Asphalt und Asphaltstrassen"; 1888; Liter. artistische Anstalt; Munich.

Schulberg, Karl; "Der Waldwegbau und seine Vorarbeiten" (Vols. I and II); 1873-5; Verlag von Julius Springer; Berlin.

Shaler, N. S.; "American Highways; A Popular Account of their Condition"; 1896; The Century Co.; New York.

Sille, N.; "Leitfaden für Strassenbau und Strassenerhaltung"; 1917; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Simms, F. W.; "Practical Observations on the Asphaltic Mastic, or Cement of

Seyssel now Extensively Employed on the Continent for Pavements; Roofing and Flooring, for Hydraulic Works, etc."; 1837; J. Weale; London.

Sinsabaugh, L. W.; "Digest of United States Patents for Paving and Roofing Compositions to Jan. 1, 1875, and English Paving Compositions to Jan. 1, 1875"; 1875; U. S. Government Printing Office; Washington, D. C.

Siwel, W. H.; "Das erste techecho-slowakische Strassenbauhandbuch"; 1932; Verlag der Redaktion und Administration des Strassenbauhandbuches; Prag.

Smith, J. Walker; "Dustless Roads Tar Macadam"; 1909; Chas. Griffin & Co., Ltd.; London.

Spalding, F. P.; "Text-Book on Roads and Pavements"; 1st Edition, 1894; 2nd Edition, 1902; 3rd Edition, 1908; 4th Edition, 1912; 4th Edition, Revised, 1913; John Wiley & Sons, Inc.; New York.

Spielmann, P. E. and *Elford, E. J.;* "Road Making and Administration" (Vol. I of The Roadmakers' Library); 1934; Edward Arnold & Co.; London.

Spielmann, P. E. and *Hughes, A. C.;* "Asphalt Roads" (Vol. V of The Roadmakers' Library); 1936; Edward Arnold & Co.; London.

Stellwaag, A.; "Der Kornaufbau von Schwarzstrassen"; 1936; Allgemeiner Industrie-Verlag G.m.b.H.; Berlin-Lichterfelde.

Tabary, A. R.; "Manuel pratique de l'ingénieur sur la technique de la route"; 1933; Léon Eyrolles; Paris.

Technischen Hochschule; "Mitteilungen aus der Forschungsstelle für Strassenbau an der Technischen Hochschule zu Braunschweig" (Vol. IV "Die Eignung des Steinkohlenteere zum Strassenbau"); 1931; ———; Braunschweig.

Tillson, Geo. W.; "Street Pavements and Paving Materials"; 1st Edition, 1900; 2nd Edition, 1903; 3rd Edition, 1912; John Wiley & Sons, Inc.; New York.

Todt, Fritz; "Fehlerquellen beim Bau von Landstrassendecken aus Teer und Asphalt"; 1932; Verlag von Martin Boerner; Halle (Saale).

Touring Club Italiano; "Il Laboratorio dell' Institute Sperimentale Stradale 1919-1930"; 1931; Touring Club Italiano; Milan.

Tucker, J. I.; "The American Road: A Non-engineering Manual for Practical Road Builders; 1916; Published by the Author; Norman, Oklahoma.

Umpfenbach, F. A.; "Theorie des neubaues, der Herstellung und Unterhaltung der Kunststrassen"; 1830; Rücker Verlag; Berlin.

Val de Travers Asphalte Paving Co., Ltd.; "Practical Notes and Illustrations of the Uses of Mastic Asphalts"; 1912; Val de Travers Asphalte Paving Co.; Ltd.; London.

Verband der österreichischen Strassengesellschaften; "Merkblatt, betreffend die wichtigsten Schäden auf Strassendecken, ihre Entstehung, Behebung und Verhütung"; 1936; Verband der österreichischen Strassengesellschaften; Vienna.

Verlag Gesellschaft für Strassenwesen; "Beschreibung der neuzeitlichen Strassenbauweisen"; 2nd Edition, 1935; Verlag Gesellschaft für Strassenwesen in Wien und Niederösterreich; Vienna.

Verres, Rudolf; "Die moderne Strassenbau"; 1927; Verlag von Martin Boerner; Halle (Saale).

Vespermann, Magistratsbaurat; "Teermakadam"; 1st Edition, 1926; "Teermakadam also neuzeitlicher Fahrbahnbelag"; 2nd Edition, 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Vespermann, Magistratsbaurat; "Vor- und Nachteile neuzeitlicher Strassenbefestigung sowie ihr Anwendungsgebiet"; 1928; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Vespermann, Magistratsbaurat; "Wahl der Strassenbefestigung nach wirtschaftlichen Gesichtspunkten"; 1929; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Vespermann, Magisträtsbaurat; "Verwendung von Hart- und Weich- sowie künstlichen Gesteinen bei neuzeitlichen Strassendecken"; 1934; Union Deutsche Verlagsgesellschaft; Berlin.

Vinsonneau, Jules; "La route moderne"; 1909; H. Dunod et E. Pinat; Paris.

Wakelam, H. T.; "Practical Road Work"; 1916; St. Bride's Press; London.

Wattebled, F. and *Carini, F.;* "Construction de la route"; 1929; Ch. Béranger; Paris.

Wattebled, F. and *Carini, F.;* "La brique de pavage"; 1929; Ch. Béranger; Paris.

Weiss, Friedrich; "Die Konstruktion neuzeitlicher Strassendecken" (Vol. 34 "Technische Fortschrittsberichte"); 1934; Verlag von Theodor Steinkopff; Dresden and Leipzig.

Weicht, A. H.; "Bau von Strassen- und Strassenbahnen"; 1902; Verlag von M. Weicht-Steglitz; Berlin.

Westmeyer, R.; "Ueber die Verwendung von Verschnitt-bitumen im Praktischen Strassenbau und die ihre Beschaffenheit und Bewährung zu stellenden Anforderungen"; 1934; Verlag von Martin Boerner; Halle (Saale).

Wheeler, W. H.; "The Repair and Maintenance of Roads"; 1900; Longmans, Green & Co. Ltd.; London and New York.

Whinery, Samuel, "Specifications for Street Roadway Pavements"; 1st Edition, 1907; Engineering News Publishing Co.; New York; 2nd Edition, 1913; McGraw-Hill Book Co., Inc.; New York.

Whyatt, H. Gilbert; "Streets, Roads and Pavements"; 1923; Sir Isaac Pitman & Sons; New York.

Wieland, G. and *Stöcke, K.;* "Merkbuch für Strassenbau"; 1934; Verlag von Wilhelm Ernst & Sohn; Berlin.

Wild, L.; "Der Strassenstaub und dessen Bekämpfung"; 1911; Anton Seydel Verlag; Berlin.

Wiley, C. C.; "Principles of Highway Engineering"; 1st Edition, 1928; 2nd Edition, 1935; McGraw-Hill Book Co., Inc.; New York.

Wilhelmi, R.; "Neue Wege für den Aufbau von Asphalt- und Teerdecken" (Mitteilungen der Versuchsanstalt für Strassenbau, Techn. Hochschule Stuttgart, Vol. 7); 1933; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Willcocks, G. W.; "Roads and Roadways"; 1879; E. F. & N. Spon, Ltd.; London.

von Willmann, Leo; "Strassenbau"; 1895; Verlag von Wilhelm Engelmann; Leipzig.

Wlassics, Hans; "Jahrbuch für das Strassenbauwesens in Oesterreich für 1931; Published 1931; Same for 1932, Published 1932; Same for 1933, Published

1933; Same for 1934, Published 1934; Same for 1935, Published 1935; In Selbstverlage des Verfassers; Linz, Austria.

Woernle, E.; "Geräte und Maschinen des nordamerikanischen Landstrassenbaues"; 1926; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin; Zementverlag; Charlottenburg.

Wood, Francis; "Modern Road Construction"; 1st Edition, 1912; 2nd Edition, 1920; Charles Griffin Co., Ltd.; London.

(I) SUNDRY BITUMINOUS COMPOSITIONS

Badder, H. C.; "Impervious Concrete"; 1923; The Education Publishing Co., Ltd.; London.

Barry, T. H., Drummond, A. A. and *Morrell, R. S.;* "The Chemistry of the Natural and Synthetic Resins"; 1926; Ernest Benn, Ltd.; London.

Bell, L. M. T.; "The Making and Moulding of Plastics"; 1936; Hutchinson's Scientific & Technical Publications; London.

Bottler, Max.; "Kunstharzen"; 1919; J. F. Lehmann's Verlag; Munich.

Brandenburger, Kurt.; "Herstellung und Verarbeitung von Kunstharz-Pressmassen"; Vol. I, 1934; Vol. II, 1935; Vol. III, 1936; Vol. IV, 1937; J. F. Lehmann's Verlag; Munich.

Dagnelies, Maur; "L'enrobage des pierrailles naturelles au moyen d'émulsion de bitume, bitume fluxé, bitume-goudron"; 1935; De Vos-Van Kleef; Antwerp.

Ellis, Carleton; "Synthetic Resins and their Plastics"; 1923; The Chemical Catalog Co., Inc.; New York.

Escard, Jean; "Les Substances Isolantes"; 1911; Gauthier-Villars et Cie.; Paris.

Fischer, Emil J.; "Kitt-Wegweiser"; 1931; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Fritsch, J.; "Fabrication des matières plastiques"; 1926; Librairie Centrale des Sciences Desforges, Girardot & Cie.; Paris.

Grafe, Victor; "Grafe's Handbuch der organischen Warenkunde" (Vol. V. "Borsten, Schwämme, Plastische Massen, Federn, Synthetische Produkte, Heilmittel, Künstliche Gerbmittel"); 1929; Verlag von C. E. Poeschel; Stuttgart.

Hemming, Emile; "Plastics and Molded Electrical Insulation"; 1923; The Chemical Catalog Co., Inc.; New York.

Höfer, Johannes; "Die Fabrikation künstlicher plasticher Massen"; 3rd Edition, 1908; A. Hartleben's Verlag; Vienna and Leipzig.

Karo, W.; "Kunstharze"; 1932; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Krausch, Oskar; "Handbuch der künstlichen plastischen Massen"; 1931; J. F. Lehmann's Verlag; Munich.

Lehner, Siegmund; "Die Kitte und Klebmittel"; 8th Edition, 1916; *Lehner, Siegmund,* and *Wächter, Friedrich;* 9th Edition, 1922; 10th Edition, Revised, 1933; A. Hartleben's Verlag; Vienna and Liepzig.

Main, W., and *Chaplet, A.;* "Toutes les matières plastiques artificielles"; 1936; Librairie des Sciences Pratiques Desforges; Paris.

Morrell, R. S.; "The Chemistry of Synthetic Resins and Allied Plastics"; 1932; Ernest Benn, Ltd.; London.

Nouvel, Otto; "Die Industrie der Phenol-Aldehyd-Harze" (Vol. XLVII Monographien über chemischtechnische Fabrikationsmethoden); 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Poirson, L.; "Chaux, ciments, platre, bitumes et asphaltes, mortiers et bétons; fabrikation, propriétés, emploi"; 1931; S. F. d'Editions Littéraires et Techniques; Edgar Malfère; Paris.

Rahm, Louis F.; "Plastic Molding"; 1933; McGraw-Hill Book Company, Inc.; New York and London.

Rowell, H. W.; "The Technology of Plastics"; 1936; Plastics Press, Ltd., London.

Scheiber, Johannes and *LeBlanc, Max.;* "Kunststoffe"; 1934; Akademische Verlagsgesellschaft; Leipzig.

Scheiber, Johannes and *Sändig, Kurt;* "Die künstlichen Harze"; 1929; Wissenschaftliche Verlagsgesellschaft m.b.H.; Stuttgart.

Scheiber, Johannes and *Sändig, Kurt;* "Artificial Resins" (Translated from the German by Ernest Fyleman); 1932; Sir Isaac Pitman & Sons; New York.

Sommerfeld, Arthur; "Plastische Massen: Herstellung, Verarbeitung und Prüfung nicht metallische Werkstoffe für spanlose Formgebung"; 1934; Verlag von Julius Springer; Berlin.

Tede, Alfons; "Kitte und Klebstoffe"; 1935; Verlag für chemische Industrie; Augsburg.

(J) ROOFING AND WATERPROOFING PRODUCTS

Abraham, Herbert; "Shelter; A Brief Glimpse at the Homes of Men Down through the Ages"; 1936; Published by the Author; New York.

Alfeis, Carl; "Untersuchungen über Ursachen der Zerstörung von Grundwasser-Isoliermaterialien"; 1931; Druckerei Gutenberg; Braunschweig.

Andés, L. E.; "Feuerischer-, Geruchlos- und Wasserdichtmachen aller Materialien" (Vol. 222); 2nd Edition, 1922; A. Hartleben's Verlag; Vienna and Leipzig.

Andés, L. E.; "Papier-Specialitäten" (Vol. 223); 2nd Edition, 1922; A. Hartleben's Verlag; Vienna and Leipzig.

Anonymous; "Asphalt-Ciment; Anleitung zur richtigen und zweckmäszigen Verarbeitung des Asphalts zu Fuszböden, Troittoirs, Fahrwegen, Dachdeckungen, usw."; 1839; Mittler; Berlin.

Beck, Joseph; "Anleitung zum Eindeckung der Dächer mit Steinpappe aus der Fabrik wasserdichter Stoffe des Verfassers"; 1859; Finsterlin Verlag; Munich.

Benicke, Anton; "Die in neuerer Zeit in Vorschlag gebrachten leichteren und minderkostspieligen Dachdeckungen von Zink, Guszeisen, Holz, Pappe, usw."; 1832; Basse; Leipzig (Quedlinberg).

Bergwald, Fritz; "Grundwasserdichtungen"; 1916; Verlag von R. Oldenbourg; Munich.

Blake, Ernest G.; "Damp Walls"; 1923; D. Van Nostrand Co. Inc.; New York.
Blake, Ernest G.; "Roof Coverings"; 1925; D. Van Nostrand Co. Inc.; New York.
Böthke, E. H. S.; "Anweisung zum Bau mit getrockneten Lehmziegeln, nebst Beschreibung des gestreckten Winkelbodens, und die besten Art Dächer"; 1808; W. G. Korn; Breslau.
Bottcher, Friedrich Wilhelm; "Abhandlung über die Construktion der Dächer und deren Bedeckung auf den ländlichen Gebäuden"; 1815; Wanderhöck u. R.; Göttigen.
Böttiger, Max; "Die Dachdecker auf dem Lande"; 1862; Verlag von E. Schotte & Co.; Berlin.
Bottler, Max; "Technische Anstrich–, Imprägnier– und Isoliermittel"; 1921; Verlagsdruckerei Würzburg, G.m.b.H.; Würzburg.
Büsscher and *Hoffman, G.m.b.H.;* "Anweisung zum Eindecken der Dächer mit Steinpappe"; 1857; Büsscher and Hoffman, G.m.b.H.; Berlin.
Buttel, Friedrich; "Praktische Erfahrungen über Dorn'sche Dächer, nebst Anhang über Anwendung der flachen Dächer"; 1841; G. Barnewitz; Neubrandenburgh and Friedland.

von Dalberg, Carl; "Versuch einiger Beyträge über die Baukunst"; 1792; Keyser; Erfurt.
David, Luzian; "Neuzeitliche freitragende Dacheindeckungen"; 1927; Verlag von Julius Springer; Berlin.
Denfer, J.; "Couverture des édifices; ardoises, tuiles, métaux, etc." (Architecture et constructions civiles); 1893; Gauthier-Villars et Cie.; Paris.
Dorn, J. F.; "Praktische Anleitung zur Ausführung der neuen flachen Dachdeckungen, Anlegung künstlichen Fuszwege, usw."; 2nd Edition, 1835; 3rd Edition, 1838; Schröder; Berlin.
Dorn, J. F.; "Anweisung zum Bau der Dorn'schen Lehmdächer"; 1836; Schröder; Berlin.
Dörr, Clemens; "Die Uebergriffe der Heimatschutzbestrebungen"; 1868; Druck der Hofbuchdruckerei G. Pflaum; Wiesbaden.
Duhamel, du Monceau; "Art du couvreur"; 1766; Imprimerie de L. F. Delatour; Paris.
Duhamel, du Monceau; "Die Kunst des Dachdeckers"; 1767; Scheber; Königsberg.
Düwer, Branddirektor; "Feuersicherheit des Pappdaches"; 1931; Verlag von Wilhelm Knapp; Halle (Saale).

Ebart, Gebr.; "Anleitung zum Bau der Pappdächer"; 1852; ———; Berlin.
Elben, Alfred; "Die Fabrikation der teerfreier Dachpappen"; 1924; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.
Elben, Alfred; "Die Fabrikation farbiger teerfreier Dachpappen"; 1925; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Falian, C.; "Vedag-Buch"; 1st Edition, 1928; Published yearly thereafter; 9th Edition, 1936; Vereinigte Dachpappenfabriken A.-G.; Berlin.
Faxe, Arvid; "Inträdes-Tal om. Sten-Papper Hallet, fòr Kongl. Vetenskaps Academien"; 1787; Kgl. Akademie der Wissenschaften; Stockholm.

Feltone, Eduard; "Isoliermaterialien und Wärme– (Kälte–) Schutzmassen" (Vol. 263); 1903; A. Hartleben's Verlag; Vienna, Budapest and Leipzig.

Gilly, David; "Beschreibung der Feuer abhaltenden Lehmschindel Dächer; 1796; Verlag Friedrich Maurer; Berlin.
Gilly, David; "Ueber Erfindung, Construktion und Vortheile die Bohlendächer; 1797; Verlag von Friedrich Vieweg & Sohn; Braunschweig.
Gilly, David; "Anweisung zur Anwendung der Bohlen-Dächer bey ökonomischen Gebäuden"; 1801; Verlag Friedrich Maurer; Berlin.

Helfenzrieder, J. E.; "Beschreibung eine neuen Art Dachziegel"; 1786; Duyle; Salzburg.
Herzberg, Friedrich; "Vorschläge zur Verbesserung der bisher üblichen Dächer"; 1774; W. G. Korn; Breslau.
Hoyer, Fritz; "Die Dachpappenfabrikation"; 1927; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.

Jeep, Wilhelm; "Die Eindeckung der Dächer mit weichen und harten Materialien, namentlich mit Stein, Pappe und Metall"; 1st Edition, 1882; 2nd Edition, 1892; Verlag von Bernhard Friedrich Voigt; Leipzig and Weimar.

Keim, A. W.; "Die Feuchtigkeit der Wohngebäude" (Vol. 89); 2nd Edition, 1901; A. Hartleben's Verlag; Vienna, Budapest and Leipzig.
Keim, A. W.; "The Prevention of Dampness in Buildings" (Translated from the German); 1902; Scott, Greenwood & Son; London.
von Kfeller; "Neue, einfache und dauerhafte Dachdeckungsart"; 1799; Eggenberger; Prag, Krappe, Leipzig.
Koetz, Arthur; "25 Jahre Verband Deutscher Dachpappenfabrikanten"; 1936; Buchdruckerei des Waisenhauses; Halle (Saale).
Koller, Theodor; "Die Imprägnierungs-Technik"; 1st Edition, 1896; 2nd Edition (2 Vols.), 1923; A. Hartleben's Verlag; Vienna, Budapest and Leipzig.

Lange, Joh. Gottfr.; "Abhandlung über wetterfeste Dächer"; 1785; Schneider; Leipzig.
Leo, Wilhelm; "Die Dachpappe, deren Haltbarkeit und Wert als Bedachungsmaterial, deren Herstellung im Kleinen und Groszen"; 1858; Basse; Quedlinburg.
Lewis, Myron H.; "Modern Methods of Waterproofing"; 1914; Norman W. Henley Publishing Co.; New York.
Liesheim, Otto; "Zum Wiederaufbau Ostpreuszens—Das flache Dach"; 1915; Allgemeiner Industrie-Verlag, G.m.b.H.; Berlin.
Linke, Gustav; "Die Bau der Dornschen Lehmdächer"; 1837; Verlag von Friedrich Vieweg & Sohn; Braunschweig.
Linke, Gustav; "Der Bau der flachen Dächer unter Benutzung von Lehm, Pappe, usw."; 1840; Verlag von Friedrich Vieweg & Sohn; Braunschweig.
Linke, Gustav; "Die Dachnoth"; 1841; Verlag von Wilhelm Knapp; Halle (Saale).
Luhmann, E.; "Die Fabrikation der Dachpappe und der Anstrichmasse für Pappdächer, in Verbindung mit der Teerdestillation, nebst Anfertigung aller Arten von Pappebedachungen und Asphaltierungen"; 1st Edition, 1883;

2nd Edition, 1902; *Luhmann, E.* and *Eszlinger, R.;* 3rd Edition, 1929; A. Hartleben's Verlag; Vienna, Budapest and Leipzig.

Luppe, Theodor; "Die moderne Bedachungen – das Rasendach und die Deckung mit Holzzement"; 1869; Dominicus; Prag.

Malchow, Wolfgang and *Mallison, Heinrich;* "Die Industrie der Dachpappe"; 1928; Verlag von Wilhelm Knapp; Halle (Saale).

Marperger, P. J.; "Altanen – Eine Werbeschrift für das flache Dach aus dem 18. Jahrhundert"; 1722; Republished 1930; v. Friedrich Bock and G. G. Wieszner, Verlag Ernst Frommann & Sohn; Nürnberg.

Mellin, J. F. C.; "Anweisung zu eine neuen, wohlfeilen, dauerhaften und feuerabhaltenden Dachdeckung auf Landgebäuden"; 1828; Schüppel; Berlin.

Mercer, C. D.; "What is Your Market – an Analysis of the Roofing Situation"; 1916; Curtis Publishing Co.; Philadelphia, Pa.

Michaut, C. F.; "Praktische Erfahungen welche ich in meinem Hause bei Anlegung eines flachen Lehmdaches zu machen Gelegenheit hatte"; 1837; Albert Förstner; Berlin.

Mierzinski, Stanislaus; "Handbuch der praktischen Papier-Fabrikation" (Vols. I, II and III); 1886; A. Hartleben's Verlag; Vienna and Leipzig.

Mierzinski, Stanislaus; "The Waterproofing of Fabrics" (Translated from the German by Arthur Morris and Herbert Robson); 1st Edition, 1903; 2nd Edition, 1914; 3rd Edition, 1921; Scott, Greenwood & Co.; London.

Northwestern Lumbermens' Association; "Ready Roofing Mists and Mysteries"; 1911; Northwestern Lumbermens' Association; Minneapolis, Minn.

Nünnecke, C. L.; "Anweisung zur Ausführung feuersicherer Bedachung von Lehm und Teer, usw."; 1839; Hendesz; Cöslin.

Odinga, Theodor; "Die Kiesklebedächer, ihre Ausführung und ihre Feuersicherheit"; 1916; Selbstverlag des Verbandes schweizerische Dachpappenfabrikanten; Zürich.

Odinga, Theodor; "Die Dachpappdächer und ihre Feuersicherheit"; 1916; Selbstverlag des Verbandes schweizerische Dachpappenfabrikanten; Zürich.

Opderbecke, Adolf; "Der Dachdecker und Bauklempner"; 1st Edition, 1900; 2nd Edition, 1907; 3rd Edition, 1921; Verlag von Bernhard Friedrich Voigt; Leipzig.

Opderbecke, Adolf; "Die Dachschiftungen"; 3rd Edition, 1920; Verlag von Bernhard Friedrich Voigt; Leipzig.

Opderbecke, Adolf; "Dachausmittelungen"; 2nd Edition, 1921; Verlag von Bernhard Friedrich Voigt; Leipzig.

Pearson, Herbert P.; "Waterproofing Textile Fabrics"; 1924; The Chemical Catalog Co., Inc., New York.

Peterson-Kinberg, Willy; "Wie eine moderne Teerdestillation mit Dachpappenfabrik eingerichtet sein muss"; 1904; A. Hartleben's Verlag; Vienna and Leipzig.

Plagge, Theodorich; "Die Fabrikation von Dachpappe-Gewerbeblatt fur däs Groszherzogtum Hessen"; 1861; Heuser; Darmstadt.

Prepared Roofing Association; "New Uses for Prepared Roofing"; 1923; Prepared Roofing Association; Chicago, Ill.

Recordon, Benjamin; "Das Ziegeldach in seiner historischen und technischen Entwicklung"; 1915; Emil Pfenninger & Co.; Zürich, Switzerland.

Reichsverband deutscher Dachpappenfabrikanten E. V.; "Merkblätter für die Herstellung von Pappdächern"; 1932; Reichsverband deutscher Dachpappenfabrikanten e.V.; Berlin.

Reimann, E. J.; "Verfahren beim Legen der Pappdächer"; 1854; Leuckart; Breslau.

Ross, Joseph; "Waterproofing Engineering"; 1919; John Wiley & Sons, Inc.; New York.

Rüber, Eduard; "Das Rasendach: die wohlfeilste, dauerhafteste und feuersicherste Eindeckungsart für Stadt- und Land- Gebäude"; 1860; Literar.-artistische Anstalt; Munich.

Runge, F. F.; "Das flache Lehmdach und der elastische Theerfirniz"; 1837; Sander; Berlin.

Sachs, Salomon; "Beschreibung einer neuen Dachkonstruktion, in welcher durch Kostenanschläge die vorteilhafte Anwendung zu Scheunen dargetan wird"; 1829; Albert Förstner; Berlin.

Sachs, Salomon; "Beschreibung einer neuerfundenen Dachkonstruktion; wasserdicht, feuerfest, usw."; 1829; Schüppel; Berlin.

Sachs, Salomon; "Beschreibung einer neuerfundenen Dachkonstruktion zu städtischen und ländichen Gebäuden aller Art anwendbar"; 1829; Albert Förstner; Berlin.

Sachs, Salomon; "Anweisung zur Anfertigung einer neuen, völlig feuerfesten, und absolut wasserdichten Dachbedeckung für ganz flache Dächer (Altane), mittels eigens darzu erfundener Harzplatten"; 1837; Albert Förstner; Berlin.

Sachs, Salomon; "Der Lehmbau, durchaus verbessert und bewährt durch 20-jahrige praktische Erfahrung, nebst der doppelten Harzplattendeckung"; 1841; Albert Förstner; Berlin.

Scherer, Robert, "Die künstlichen Fuszboden- und Wändebeläge"; 1st Edition, 1907; "Die künstlichen Fuszböden-, Wändebeläge und Deckenverkleidungen"; 2nd Edition, 1922; A. Hartleben's Verlag; Vienna and Leipzig.

Schmidt, Otto; "Die Eindeckung der Dächer und die Konstruktion der Dachrinnen mit Berücksichtigung aller neueren Erfahrungen und Erfindungen"; 1885; Verlag Costenoble; Jena.

Schmiedel, Ottomar; "Die Scheddachbauten"; 1904; W. S. Loewenthal; Berlin.

von Schönberg, Alex; "Die Pappdächer. Erfahrungen über Haltbarkeit und Werth, sowie Anleitung zur Anfertigung derselben"; 1st and 2nd Editions, 1857; Schönfeld; Dresden.

von Schönfeld, J. F.; "Die Kunst das unverbrennbare Steinpapier zum Dachdecken nachzuahmen"; 1792; Wimmer; Vienna.

Schöning, von A.; "Versuch einer feuersicheren und wohlfeilen Bedachung länd Gebäude"; 1830; Lüderitz; Berlin.

Schrader, Fritz and *Reim, H.;* "Der deutsche Dachdeckermeister"; 1911; Verlag von Wilhelm Engelmann; Leipzig.

Schultze, Joachim; "Grundwasserabdichtung"; 1919; Verlag von Wilhelm Ernst & Sohn; Berlin.

Sesselberg, Friedrich; "Das Flache Dach im Heimatbilde"; 1912; Verlag von Weise & Co.; Berlin.

Shank, J. J.; "The Analysis and Testing of Roofing Surfacing Materials and Composition Roofing"; 1935; R. J. Funkhouser & Co. Inc.; Hagerstown, Md.

Sinsabaugh, L. W.; "Digest of United States Patents for Paving and Roofing Compositions to Jan. 1, 1875, and English Paving Compositions to Jan. 1, 1875"; 1875; U. S. Government Printing Office; Washington, D. C.

Steiner, J. F. R.; "Entwurf einer neuen und durchaus feuerfesten Bauart mit gewölbten Decken und Dachungen zur Sicherheit und Wohlfahrt der menschlichen Wohnungen und anderer Gebäude"; 1803; Hoffman; Weimar.

Verband deutscher Dachpappenfabrikanten and Staatlichen Materialprüfungsamtes; "Vergleichende Versuche mit Rohteer, destilliertem und präpariertem Teer und Prüfungen von Roh- und Dachpappe"; 1924; Verlag von Wilhelm Knapp; Halle (Saale).

Verband deutscher Dachpappenfabrikanten and staatlichen Materialprüfungsamtes; "Vergleichende Versuche mit Teerdachpappen sowie den zugehörigen Rohstoffen"; 1925; Verlag von Wilhelm Knapp; Halle (Saale).

Verband deutscher Dachpappenfabrikanten and staatlichen Materialprüfungsamtes; "Vergleichende Versuche mit teerfreien Dachpappen sowie den darzugehörigen Rohpappen, Tränk- und Belagmassen"; 1925; Verlag von Wilhelm Knapp; Halle (Saale).

Wandrowsky, Hermann; "Wasserdichtmachen von Papier"; 1927; Verlag der Papier-Zeitung, Carl Hofmann, G.m.b.H.; Berlin.

Weiher, H.; "Richtig Isolieren"; 1930; Verlag Dr. Fritz Wedekind & Co.; Stuttgart.

Weniger, K. A.; "Die Herstellung von Asbestpappe und Asbestpapier" (Vol. 362); 1920; A. Hartleben's Verlag; Vienna and Leipzig.

Wernicke, K.; "Die Isoliermittel der Electrotechnik"; 1911; Verlag von Friedrich Vieweg & Sohn; Braunschweig.

Whitehead, J. B.; "Impregnated Paper Insulation"; 1935; John Wiley & Sons, Inc.; New York.

Winkelmann, Architekt; "Anweisung zur Ausfertigung der Dächer aus Lehm und Steinkohlentheer nach Angabe des Fabriken-Commissionsrathes Dorn, und nach eigenen Erfahrungen bearbeitet"; 1835; Logier; Berlin.

REFERENCES

CHAPTER I

1 [p. 2] "Notes on the Pleistocene Fossils Obtained from Rancho La Brea Asphalt Pits," by L. E. Wyman, Miscellaneous Publication No. 2, Los Angeles County Museum of History, Science and Art; Los Angeles, California (May, 1918).

2 [p. 5] "Occurrence of Pleistocene Vertebrates in an Asphalt Deposit near McKittrick, California," by J. C. Merriam and Chester Stock, Science, **54**, 566 (1921).

3 [p. 5] "Aus der ältesten Geschichte des Bitumens," by R. J. Forbes, Bitumen, **4**, Nos. 1, 2 and 3 (1934); "Aspects of Sumerian Civilization as Evidenced on Tablets in the John Rylands Library—1. About Building in Ur III," by T. Fish; Bull. John Rylands Library, **18**, No. 1, 134 (1934); "Das Bitumen in den fünfzehn Jahrhunderten vor Drake (300–1860)," by R. J. Forbes, *Bitumen*, **7**, 11, 37 and 59 (1937).

4 [p. 5] "Cuneiform Parallels to the Old Testament," by R. W. Rogers, pp. 91 and 135, Eaton & Mains, New York (1912); "Myths of Babylonia and Assyria," by D. A. Mackenzie, p. 191, Gresham Publishing Co., London (1916).

5 [p. 5] "Mémoires de la Délégation en Perse," published under the direction of M. J. de Morgan, Vol. II (1900), Plate IX; Vol. VIII, p. 162; Vol. X (1908), Plate I; Vol. XIII, Plates XXXIII and XXXVI, published by Ernest Leroux, Paris; also Comptes Rendus de l'Académie d'Inscriptions, pp. 398–399, Figs. 1 and 2, by M. J. de Morgán (Jul., 1907).

6 [p. 7] "An Asphalt Ring from Ur of the Chaldees," by J. E. Hackford, S. Lawson and P. E. Spielmann, *J. Inst. Petrol. Tech.*, **17**, 738 (1931); "Note on a Lump of Asphalt from Ur," by R. J. Forbes, *J. Inst. Petrol. Tech.*, **22**, 180 (1936).

7 [p. 7] "Ur Excavations," by H. R. Hall, Vol. I, "Al-'Ubaid (El Obeid)," Oxford (1927); "Ur und die Sintflut," by C. L. Wooley, F. A. Brockhaus, Leipzig and Stuttgart (1932).

8 [p. 8] "Tell Asmar and Khafaje—The First Season's Work in Eshnunna 1930–31," by Henri Frankfort, Thorkield Jacobsen and Conrad Preusser, Univ. of Chicago Oriental Inst. Communication No. 13, Univ. of Chicago Press, Chicago, Ill., (1932); "Tell Asmar, Khafaje and Kharsabad—Second Preliminary Report of the Iraq Expedition," by Henri Frankfort, Univ. of Chicago Oriental Inst. Communication No. 16, Univ. of Chicago Press, Chicago, Ill. (1933); "Iraq Excavations of the Oriental Institute 1932/33—Third Preliminary Report of the Iraq Expedition," by Henri Frankfort, Univ. of Chicago Oriental Inst. Communication No. 17, Univ. of Chicago Press, Chicago, Ill. (1934).

9 [p. 10] "Vor 5000 Jahren," by C. L. Wooley, Leipzig (1930); "Ur und die Sintflut," by C. L. Wooley, F. A. Brockhaus, Leipzig and Stuttgart (1932); "Untersuchungen Ueber die ältesten Anwendungen von Bitumen in Mesopotamien," by R. J. Forbes, Bitumen, **5**, 9, 41 and 63 (1935); "Asphalt Found in Mesopotamian Excavations," by F. J. Nellensteyn and J. Brand, *Chem. Weekblad*, **33**, 261 (1936); "Het Bitumen in de Verfindustrie," by R. J. Forbes, Verfkroniek, **9**, 263 (1936).

10 [p. 10] "The Civilization of Babylonia and Assyria," by Morris Jastrow, Jr., pp. 394–395; J. B. Lippincott Co., Philadelphia (1915).

11 [p. 10] "Bismya, or the Lost City of Adab," by E. J. Banks, G. P. Putnam's Sons, New York, pp. 191, 271 and 314 (1912).

12 [p. 12] Foundation Eugène Piot, "Monuments et Mémoires," by Ernest Leroux, Paris, Vol. VI (1899) and Vol. VII (1900).

13 [p. 12] Foundation Eugène Piot, "Monuments et Mémoires," by Ernest Leroux, Paris, Vol. I (1894).

14 [p. 12] "Mémoires de la Délégation en Perse," published under the direction of M. J.

de Morgan, Vol. XIII, "Récherches Archèologiques Cinquième Série de l'Epoque Archaique," by Edm. Pottier, published by Ernest Leroux, Paris (1912).

15 [p. 13] "Mémoires de Henri Le Chatelier," Vol. VIII, p. 162, A. Colin, Paris (1913).

16 [p. 13] "Relics of Remote Antiquity from Ur," by C. L. Wooley, Illustrated London News, p. 327 (Mar. 1, 1930).

17 [p. 14] "Découvertes en Chaldée"; also "Catalogue des Antiquités Chaldéennes du Musée National du Louvre," by Henzey, Paris, No. 125 (1902).

18 [p. 14] "Die Sumerischen und Akkadischen Königsinschriften," by François Thureau-Dangin, J. C. Heinrichs Verlag, Leipzig (1907).

19 [p. 14] "Myths of Babylonia and Assyria," by D. A. Mackenzie, p. 191, Gresham Publishing Co., London (1916).

20 [p. 15] "Das Steinbuch aus der Kosmographie des Zakarija ibn Muhammad ibn Mahmud al-Kazwînî," by J. Ruska, Beil. zum Jahresber. Oberrealschule Heidelberg, p. 43 (1895–96).

21 [p. 15] "Indus Civilization," by J. Marshall, Archaeol. Survey India, Annual Reports 1923–1924 and 1925–1926; "Mohenjo-Daro and Indus Civilization," by J. H. Marshall; A. Probsthain, London (1931).

22 [p. 15] "Examination of Asphalt Found in Excavations at Mohenjo-Daro (Indus Valley)," by F. J. Nellensteyn, *Chem. Weekblad*, 31, 543 (1934); Bitumen, 5, 15 (1935).

23 [p. 16] "A History of Egyptian Mummies," by T. J. Pettigrew, London (1834); "Persien das Land und seine Bewohner," by J. E. Polak, Vol. II, p. 274, Brockhaus, Leipzig (1865); "Preservative Materials Used by the Ancient Egyptians in Embalming," by Alfred Lucas, Survey Paper No. 12, Minister of Finance, National Printing Dept., Cairo, Egypt (1911); *J. Egypt. Archaeology*, 1, 241 (1914); "History of Assyria," by A. T. Olmstead, Chas. Scribner's Sons, New York (1923); "Wonders of the Past", by J. A. Brendon, G. P. Putnam's Sons, New York (1924); "Egyptian Mummies," by Smith and Dawson, The Dial Press, New York (1924); "The Mummy," by E. A. Wallis-Budge, Cambridge University Press, London (1925); "Vom Asphalt über die Mumie zum Ichtyol," by Fr. Netolitzky, *Pharmac. Zentralhalle*, 68, No. 4 (1927); "Un vieux remede: La Mumie," by R. Pomet, Aesculape, Paris, p. 206 (1927); "Mistakes in Chemical Matters in Archaeology," by Alfred Lucas, *J. Egypt. Archaeology*, 10, 132 (1932); "To What Extent did the Ancient Egyptians Employ Bitumen for Embalming?", by P. E. Spielmann, *J. Egypt. Archaeology*, 13, 177 (1933); "Het Egyptische Mummieportret," by W. D. van Wyngaarden, Elseviers' Maandschrift, VII, 16 (1933); "Ancient Egyptian Materials and Industries," by Alfred Lucas, 2nd Edition, Longmans, Green & Co., New York (1934).

24 [p. 17] "Geographica," by Strabo, edited by A. Meincke, Leipzig, Vol. XIV, ii, 45 (1853).

25 [p. 18] "Aus der ältesten Geschichte des Bitumens", by R. J. Forbes, *Bitumen*, 4, Nos. 1, 2 and 3 (1934).

26 [p. 18] "Relation de l'Egypte," p. 273, Edition De Sacy, Paris (1810).

27 [p. 19] "The Mummy," by E. A. Wallis-Budge, pp. 173 and 184, Cambridge University Press (1893).

28 [p. 19] "Natural Rock Asphalts and Bitumens," by Arthur Danby, p. 41, D. Van Nostrand Co., New York (1913).

29 [p. 19] "De l'embaument avant et après Jésus Christ," by L. de R. Reutter, Paris (Neuchâtel), (1912); "Ueber die in 1. Jahrtausend vor Chr. bei der Einbalsamierung in Aegypten und Kathargo benutzten Harze," by A. Tschirsch, *Archiv. für Pharmazie*, 170 (1912).

30 [p. 20] "Septuaginta Interpretes," by L. F. A. C. von Tischendorf, F. A. Brockhaus, Leipzig (1860).

31 [p. 20] "History of Assyria," by A. T. Olmstead, Chas. Scribner's Sons, New York (1923); "The Citroën-Haardt Trans-Asiatic Expedition Reaches Kashmir," by M. O. Williams, *The National Geographic Magazine*, 60, 402 (Oct. 1931); "Ueber einige Anwendungen des Asphaltes in geschichtlichen Schiffsbau," by Edmund Graefe, *Asphalt u. Strassenbau.*, p. 3 (Dec. 1931).

32 [p. 20] "The Fate of Jericho as Revealed by the Spade," by J. Garstang, *Illustrated London News*, issue of Dec. 16, 1933.

33 [p. 21] "Ausgraben in Uruk 1930–1931," by J. Jordan, *Abh. Preuss. Akad. Wiss. Phil. Hist.*, Klasse 1932.

34 [p. 21] "Excavations at Babylon," by Robert Koldewey, pp. 31, 54, 113 and 138, Macmillan & Co., Ltd., London (1914); "Die Innenstadt von Babylon (Merkes)," by Oskar Reuther, J. C. Hinrichs, Leipzig (1926).

35 [p. 21] "The Monuments of Nineveh," by A. H. Layard, J. Murray, London (1849 and 1853).

36 [p. 21] "Asphalt Einst und Jetzt," by H. Gammann, *Asphalt u. Teerindustrie-Zeitung*, 28, 1340 (1928).

37 [p. 22] "Excavations at Babylon," by Robert Koldewey, p. 54, Translation by Weissbach, Macmillan & Co., Ltd., London (1914).

38 [p. 23] "Assurbanipal und seine Zeit," by Fr. Delitsch, *Der Alte Orient*, 11, 1, 24.

39 [p. 23] "The Irrigation of Mesopotamia," by Sir W. Willocks, London (1911).

40 [p. 24] "The Cradle of Civilization," by James Baikie, *National Geog. Mag.*, 29, 130 and 151 (1916).

41 [p. 25] "Nineveh and its Palaces," by Joseph Bonomi, Ingram, Cooke & Co., Ltd., London (1852); "Keilschrifttexte aus Assur verschiedenen Inhalts," by O. Schroeder, No. 1, V. 76 (1920); "The Assyrian Herbal," by R. C. Thompson, Luzac & Co., Ltd., London (1924); "On the Chemistry of the Ancient Assyrians," by R. C. Thompson, Luzac & Co., Ltd., London (1925); "Assyrian Engineering," by R. W. Parkhurst, *Civil Eng.*, 6, 345 (1932).

42 [p. 26] "Ausgrabungen in Assur," by W. Andrae, *Mitt. deutsche Orient Gesellschaft*, Nos. 26–28, 32–33, 51–52. "Die Festungswerke von Assur," by W. Andrae, *Mitt. deutsche Orient Gesellschaft*, 23, 84 (1913).

43 [p. 26] "Les Annales de Tukulti-Ninip II, roi d'Assyrie," by Vincent Scheil and J. E. Gautier, H. Champion, Paris (1909).

44 [p. 26] "Die Keilschrifttexte Sargons," by Hugo Winckler, Leipzig (1889); "Inscriptions of Western Asia," by George Rawlinson, G. P. Putnam's Sons, New York (1982); "Das wieder erstehende Babylon," by Robert Koldewey, J. C. Hinrichs, Leipzig (1913); "Excavations at Babylon," by Robert Koldewey, pp. 31, 54, 113 and 138, Macmillan & Co., Ltd., London (1914); "Das wiederwachende Babylon," by Robert Koldewey, 4th Edition, J. C. Hinrichs Buchhandlung, Leipzig (1925).

45 [p. 27] "Babylonien und Assyrien", by Bruno Meissner, 2 vols., C. Winter Verlag, Heidelberg (1920 and 1925).

46 [p. 27] "The Lake Dwellings of Switzerland and Other Parts of Europe," by Ferdinand Keller, 2nd Edition, translated by J. E. Lee, London (1878). "Pfahlbauten," by Ferdinand Keller, 8th Bericht (1879); "Urgeschichte der Schweiz," by Jakob Heierli, pp. 122 and 184, Bern (1901); "Die Pfahlbauten des Bodensees," by Georg von Troeltsch, pp. 54 and 66 (1902); "Urgeschichte," by J. M. Forrer, *Jahresbericht Schweiz*, 11, 34 (1918); "The Oldest Swiss Lake-dwellings," by Paul Vouga, Antiquity, Vol. II, p. 387, Gloucester (1928); "Zur Geschichte der Schweizer Asphaltausbeutung in Traverstal," by L. Neuburger, *Teer u. Bitumen*, 16, 237 (1931); "Die Werkzeuge des Steinzeugmenschen," by U. and L. Pfeiffer (1920).

47 [p. 27] "Historiarum," by Herodotus, Vol. I, pp. 179 and 194; Vol. IV, p. 195; and Vol. VI, p. 119; Everyman's Library Edition.

48 [p. 27] "Collection of Voyages and Travels," by George Rawlinson, Library of Oxford, London, Vol. II, p. 752 (1745); "Inscriptions of Western Asia," by George Rawlinson, p. 59, 7b, G. P. Putnam's Sons, New York (1892).

49 [p. 28] "History of Herodotus," by George Rawlinson, New York, Vol. I, p. 245 G. P. Putnam's Sons, New York (1859).

50 [p. 28] "Mesopotamian Archaeology," by P. S. P. Handcock, Macmillan & Co., Ltd., London, p. 124 (1912).

51 [p. 29] "History of the Peloponnesian War," by Thucydides, Chap. II, 77 and Chap. IV, 100, Edition of Loeb "Classical Library."

52 [p. 29] "On Airs, Waters and Places," by Hippocrates.

53 [p. 29] "Anabasis," by Xenophon, Book II, Chap. IV, Sec. 12; Edition Brownson, Loeb Classical Library.

54 [p. 29] "De Mirabilibus Auscultationibus," by Aristotle, Chapters 35, 41, 113, 116 and 127; Edition of F. Didot (1857).

55 [p. 29] "Enquiry into Plants," by Theophrastus, IX, 3, 1, Edition Hort, Loeb Classical Library, Vol. II; "Peri Lithon," by Theophrastus, Chapters 16 and 85; German translation by K. Mieleitner, "Geschichte der Mineralogie im Altertum und im Mittelalter."

56 [p. 29] "Atlas Antiquus," by E. Rich, text facing map XVIII.

57 [p. 30] "Georgics," by Vergil (Vergilius), III, 451, Edition Fairclough, Loeb Classical Library.

58 [p. 30] "Geographica," by Strabo, I, 16; III, 144; IV, 207; V, 218; VII, 316; IX, 498; XI, 518; XVI, 1, 2, 12, 738–743, and 763–4; Edition of A. Meincke, Leipzig (1853).

59 [p. 31] "Universallexikon," by J. H. Zedler, Vol. 28, Leipzig (1732–1754).

60 [p. 32] "Bibliotheca Historica," by Diodorus Siculus, Book I, t. 2, Chap. 29; Book II, Chap. 12, t. 48, Chap. 6; Book III, t. 47, Chap. 3; Book VI, t. 19, Chap. 25; Book XVI, Chap. 40; Book XIX, t. 98, Chap. 2; P. Wesseling, Amsterdam (1746); "Hist. Universe," by Diodorus Siculus, Book VI, t. 19, Chap. 25.

61 [p. 32] "Bibliotheca Historica," by Diodorus Siculus, Book XIX, t. 98, Chap. 2; P. Wesseling, Amsterdam (1746).

62 [p. 33] "De Architectura," by Vitruvius, I, 5 and 8; VIII, 3 and 8; Edition Granger, Loeb Classical Library.

63 [p. 33] "Historiae Naturalis," by Pliny the Elder, I, 99; II, 106–8, 226, 235; V, 71; VI, 99; VII, 65; XIV, 20, 122, 127, 134; XV, 8, 62; XVI, 22, 38, 52, 56, 158; XXIV, 24, 41; XXXIII, 94; XXXIV, 15, 194; XXXV, 41, 178, 182, 194; XXXVI, 141, 166; "The Historie of the World, Commonly Called the Naturall Historie of C. Plinius Secundus," by Pliny the Elder, translated into English by Philemon Holland, II, 106–8, 226, 235; V, 71; VI, 99; VII, 65; XIV, 20; XXIV, 41; XXXIV, 15, 194; XXXV, 178, 182; printed by Adam Islip, London (1634).

64 [p. 35] "Use of Oleum Asphalti in Ulcers of the Intestines," by T. Healde, London (1769).

65 [p. 35] "Bell. Judea" (Jewish Wars), by Josephus Flavius, I, 657; III, 515; IV, 476 and 481; Edition Whiston, London (1889); "Antiquities Judea" (Jewish Antiquities), by Josephus Flavius, Book I, Chapters 4 and 20; XVII, 171 and 174; Edition Thackeray.

66 [p. 35] "Life of Sylla," by Plutarch; "Life of Alexander," by Plutarch, Chaps 35–37; Edition Perrin, Loeb Classical Library.

67 [p. 36] "Historia," by Tacitus, Vol. V, 6; Edition Moore, Loeb Classical Library.

68 [p. 36] "Variae Historiae," by Aelian (Aelianus Claudius), Vol. XIII, §16; Edition Lünemann (1811); "Geographica," by Claudius Ptolemaeus, Vol. V, 15, 3; "Technologie und Terminologie der Gewerbe und Künste bei Greichen und Römer," by Hugo Blümner, 4 Vols., Leipzig (1875–1887).

69 [p. 36] "Materia Medica," by Dioscorides, I, 83, 84, 85, 100 and 109; V, 145, 181; XIX, 98; Kühn Lipsiae (1829), Saracen (1598); "Greek Herbal," by Dioscorides, Translation by John Goodyear, edited by R. T. Gunther, Oxford University Press, Oxford (1934).

70 [p. 37] "Historiae Romana," by Dion Casius, Vol. XLI; Vol. LXVIII, 27; Vol. LXXV, 11; Edition Cary, Loeb Classical Library.

71 [p. 37] "De Vita Appolonii Tyanei" (Life of Appollonius), by Philostratus the Elder, I, 23, 24; III, 1; Edition Connybeare, Loeb Classical Library.

72 [p. 38] "Geoponica" ("Scriptores rei rusticae"), XII, 8, 1; XIII, 10, 7; XIV, 11, 4; XVII, 16, 1; XVIII, 2, 4; Edition of J. G. Schneider (1794–1797).

73 [p. 38] "Kestoi" (Treatise on agriculture, natural history, military science, etc.), by Africanus.

74 [p. 38] "Rerum Gestarum Libri," by Ammianus Marcellinus, XXIII, 4, 6, 15; XXIV, 2; XXXI; Edition C. D. Younge (1862).

75 [p. 39] "German Wild-fire Trials at Oxford in 1438," Appendix (1) "On the Sea-fire of Callinicus A.D. 673" by E. W. Hulme, Trans. Newcomen Soc., 10, 89 (1929–1930).

76 [p. 39] "Meadows of Gold and Mines of Precious Stones," by Abu-l Hasan Masudi, (950 A.D.).

77 [p. 39] "Description of the Lands of Islam," by Abd al Mukaddasi, Edition de Goeje Bibl. Geogr. Arabia, Leiden (1877).

78 [p. 40] "Chronicles of the Crusade of St. Lewis," by Pierre de Joinville, Everyman's Library Edition, pp. 186, 195 and 202.

79 [p. 40] "The Travels," by Marco Polo, Book I, Chaps. 3, 4 and 11, Everyman's Library Edition.

80 [p. 40] "Voiage and Travayle of Sir John Maundeville," Chapter XXX.

81 [p. 40] "De natura eorum quae effluent ex terrae," by Georg Agricola, Opera 1657, p. 535 (1545); "De natura fossilium," by Georg Agricola, Opera 1657, p. 595 (1546); "De re metallica," by Georg Agricola, 12th Book, Opera 1657, pp. 468 and 469, published by Basileae, Froben (1556).

82 [p. 40] "Description of Orinoco," by Gumilla (1758).

83 [p. 41] "Historia general y natural de las Indias," by G. F. Oviedo y Valdés, Toledo (1526); Modern Edition, Madrid (1853).

84 [p. 42] "The Discoverie of the Large, Rich and Bewtiful Empire of Guiana," by Sir W. Raleigh, Robert Robinson, London (1596); "Extracts from the Memoranda of the Earl of Dundonald, in the Use, Properties and Products of the Bitumen and Petroleum of Trinidad," by the Earl of Dundonald (10th), Ridgway & Co., London (1857).

85 [p. 42] "Singularium Andreae Libvaii, cont. VIII libros bituminum et affinum, historice, physice, chymice; de petroleis, ambra, halosantho, succino, gagate, asphalto, piss-asphalto, mumia, lithanthrace," by Andreas Libavius, P. Knopff, Frankfurt (1599).

86 [p. 43] "Nine Years' Travels," by Dr. J. Fryer, p. 318 et seq (1672–1681).

87 [p. 43] "Dissertation sur l'asphalte ou ciment naturel, découvert depuis quelques années au Val de Travers," by Eirinis d'Eyrinys, Paris (1721); published also as an appendix to "Der Asphalt und die Bitumina," by Josef Sporny, St. Petersburg, 2nd Edition (1878); "Histoire Naturelle des Minéraux," by Comte de Buffon, Vol. 2, L'Imprimerie Royale," Paris (1783).

88 [p. 44] "Universallexikon," by J. H. Zedler, Leipzig (1732–1754); "Altanen—Ein, Werbeschrift für das flache Dach aus dem 18. Jahrhundert," by P. J. Marperger (about 1722), reprinted by v. Friederich Bock and G. G. Wieszner, Verlag Ernst Frommann & Sohn, Nürnberg (1930); "Bitumen und Asphalt, Anno 1733," by F. M. Feldhaus, *Teer u. Bitumen*, 29, 489 (1931).

89 [p. 44] "Eléments de Minéralogie," by Le Sage, Vol. II, p. 96 (1777).

90 [p. 44] "Die Fabrikation der Dachpappe und der Anstrichmasse für Pappdächer," by E. Luhmann, p. 1, A. Hartleben's Verlag, Vienna, Budapest and Leipzig, (1883); "Inträdes-Tal om Sten-Papper Hallet, för Kongl. Vetenskaps Academien," by Naval Physician Arvid Faxe (Mar. 7, 1787); "Einige historische Notizen über Dachpappen," by Anon., *Teer u. Bitumen*, 34, 332 (1936).

91 [p. 45] "Journal für technische und Oekonomische Chemie," by O. L. Erdmann, Verlag Johann Ambrosius Barth, 6, 377 (Leipzig 1829).

92 [p. 46] Kunst-und Gewerbeblatt, p. 589 (1820).

93 [p. 46] F. Strohmeyer, *Arch. ges. Nat.*, 10, 113 (1827); J. J. Noeggerath, *J. chem. Phys.*, 63, 459 (1831).

94 [p. 46] J. J. Conybeare, *Ann. Phil.*, 21, 124 (1823).

95 [p. 46] Carl von Reichenbach, *J. Chim. phys.*, 69, 436 (1890).

96 [p. 46] **Brit. Pat.** of 1834, No. 10,327 (Sep. 26) to E. E. Cassell.

97 [p. 46] E. F. Glocker, *Schweitzerische Apotheker-Zeitung*, 69, 215 (1833); "A System of Mineralogy," by E. S. Dana, p. 998, New York (1906).

98 [p. 46] "Bitumens," by H. Wagner Halleck, p. 118, Peter Force, Washington, D. C. 1841).

99 [p. 46] "Natural Rock Asphalts and Bitumens," by Arthur Danby, p. 54, D. Van Nostrand Co., New York (1913).

100 [p. 47] "Mémoir sur la composition des bitumens," by J. B. Boussingault, *Ann. chim. phys.*, 64, 141 (1837); translated in "Asphalt Paving," by the Commissioners of Accounts of the City of New York, p. 107 (Feb. 3, 1904).

101 [p. 47] "Report on the Geological Survey of Connecticut," by C. U. Shepard, pp. 62 and 152, New Haven, Conn. (1837); "Report on the Geology of the State of Connecticut," by J. G. Percival, pp. 315, 376, 384 and 388, New Haven, Conn. (1842); "Report on the Geology of Connecticut," by J. G. Percival, *Am. J. Sci.*, 13, 130 (1842); "On Indurated Bitumen in Cavities in the Trap of the Connecticut Valley," by J. G. Percival, *Am. J. Sci.*, No. 3, 16 (1878).

102 [p. 47] "Die Chemie und Technologie der Natürlichen und Künstlichen Asphalte," by Hippolyt Köhler and Edmund Graefe, p. 21, Verlag von Friedr. Vieweg & Sohn, Braunschweig (1913).

103 [p. 47] **Brit. Pat.** of 1841 (Nov. 16), No. 9163 to Richard Gurney.

104 [p. 47] "The Occurrence of Bituminous Matter in New York State," by L. C. Beck, *Am. J. Sci.*, 14, 335 (1843).

105 [p. 47] Extracts from an autobiographical letter published by the late Reverend Samuel M. Warren, appearing in "Genealogy of the Warren Family," by Mrs. Betsy Warren Davis.

106 [p. 48] C. T. Jackson, *Proc. Boston Soc. Nat. Hist.*, p. 279 (1850); *Am. J. Sci.*, 2, XI, 292; XIII, 276 (1850).

107 [p. 48] "Asphalts, their Sources and Utilizations," by T. H. Boorman, p. 11, Wm. T. Comstock, New York (1908).

108 [p. 48] "Mineral Resources of the United States for 1893," by D. T. Day, p. 638, Washington, D. C. (1894).

109 [p. 48] J. P. Leslie, *Proc. Am. Phil. Soc.*, 9, 183 (Mar. 20, 1863).

110 [p. 48] "Natural Rock Asphalts and Bitumens," by Arthur Danby, p. 60, D. Van Nostrand Co., New York (1913).

111 [p. 49] **U. S. Pat.** 112764 of Mar. 21, 1871 to N. B. Abbott.

112 [p. 49] "Asphalts, their Sources and Utilizations," by T. H. Boorman, p. 11, Wm. T. Comstock, New York (1908).

113 [p. 49] "Mineral Resources of the United States for 1893," by D. T. Day, pp. 638–639, Washington, D. C. (1894).

114 [p. 49] "Trinidad and Bermudez Lake Asphalts," by Clifford Richardson, p. 28, Barber Asphalt Paving Co., Philadelphia (1910).

115 [p. 49] "Histoire général des choses de la nouvelle Espagne," by B. de Sahgâun, p. 630, Jourdanet & Simeon, Paris (1880); **U. S. Pat.** 1078564 of Nov. 11, 1914 to J. M. Trimble.

116 [p. 49] **U. S. Pats.** 236995 of Jan. 25, 1881 to E. J. De Smedt; 237662 of Feb. 8, 1881 to E. J. De Smedt; 239466 of Mar. 29, 1881 to E. J. De Smedt.

117 [p. 50] "Uintaite—A New Variety of Asphaltum from the Uintah Mountains, Utah," by W. P. Blake, *Eng. Mining J.*, 40, 431 (1885).

118 [p. 50] "Wurtzilite," by W. P. Blake, *Eng. Mining J.*, 48, 542 (1889).

119 [p. 50] "Die Chemie und Technologie der Natürlichen und Künstlichen Asphalte," by Hippolyt Köhler and Edmund Graefe, pp. 34–35, Verlag von Friedr. Vieweg & Sohn, Braunschweig (1913).

120 [p. 50] **U. S. Pat.** 524130 of Aug. 7, 1894 to F. X. Byerley.

CHAPTER II

1 [p. 51] "Natural Asphaltum and its Compounds," by J. H. Howard, Rensselaer Society of Engineers, Troy, N. Y. (1894); "Asphalt Paving," Report of the Commissioners of Accounts of the City of New York (Feb. 3, 1904); "Occurrence, Classification, Composi-

tion and Analysis of Asphalt," by T. B. Stillman, Stevens Institute Indicator, 389 (1904). "The Modern Asphalt Pavement," by Clifford Richardson, John Wiley & Sons, New York, p. 111 (1908); U. S. Dept. of Agriculture, Office of Public Roads, Circular No. 93 (1911); "The Question of the Nomenclature of Bitumens," by David Holde, *Petroleum*, 7, 713 (1912); "Proposals for the Definition of Bitumen," by David Holde and Julius Marcusson, *Proc. Inter. Assoc. Testing Materials*, 2, No. 1, XXV (1912); "The Classification of Bituminous and Resinous Substances," by Herbert Abraham, *J. Ind. Eng. Chem.*, 5, 11 (1913); "British Standard Nomenclature of Tars, Pitches, Bitumens and Asphalts, when Used for Road Purposes," Report of the Engineering Standards Committee, London, (Apr. 1916); "Bericht über den gegenwärtigen Stand der Nomenklaturfrage für Teere und Bitumina," by Heinrich Mallison, *Teer*, 24, 282 (1926); "Zur Frage der praktisch und wissenschaftlich richtigen Namengebung und Einteilung (Nomenklatur und Klassifizierung) für bituminöse Stoffe unter besondeher Berücksichtigung der Strassenbaustoffe," by Wilhelm Reiner, *Teer*, 24, 337 and 356 (1926); "Zur Nomenklatur der Teere und Bitumina," by Heinrich Mallison, *Teer*, 24, 357 (1926); "Eine amerikanische Stimme zur Nomenklatur der Teere und Bitumina," by Heinrich Mallison, *Teer*, 24, 508 and 574 (1926); "Teer, Pech, Bitumen und Asphalt," by Wolfgang Malchow, *Chem. Ztg.*, 53, 349 (1926); "Zur Terminologie der nicht verseifbaren, natürlichen und künstlichen Bitumina (Asphalt, Pech, Teer, usw.)," by David Holde, *Teer*, 25, 33 (1927); "Nomenklatur der Teere und Bitumina," by Heinrich Mallison, *Teer*, 25, 213 (1927); "Bitumen," by Edmund Graefe, *Asphalt und Teer Strassenbautechnik*, 31, 877 (1931); "Eine neuer Entwurf für die einheitlichen Bezeichnungen für Bitumen und verwandte Stoffe," by Anon., *Teer u. Bitumen*, 31, 189 (1933); "Standard Definitions of Terms Relating to Materials for Roads and Pavements" (D 8–33), American Soc. for Testing Materials, Standards 1936, II, 1116; "Bitumen," by Carl Engler, *Chem. Ztg.*, 36, 65 and 82 (1912).

2 [p. 58] "Immediatpeche," by E. J. Fischer, *Teer und Bitumen*, 31, 285 (1933).

3 [p. 58] "Die Industrieteere, ihre Arten, Eigenschaften und Verwendung," by E. J. Fischer, *Teer u. Bitumen*, 27, 17, 30, 69 and 125 (1929); "Künstliche Asphalte, Peche und ähnliche Produkte," by E. J. Fischer, *Teer u. Bitumen*, 28, 17 and 33 (1930); "Die chemische Beschaffenheit und Untersuchung von Teeren und Pechen," by Hans Bockshammer, *Teer u. Bitumen*, 29, 29 (1931); "Chemoasphalte und Chemopeche," by E. J. Fischer, *Teer u. Bitumen*, 33, 251 and 263 (1935); "Teerartige Rückstände," by E. J. Fischer, *Teer u. Bitumen*, 35, 123 (1937).

4 [p. 59] "Kir," by Edmund Graefe, *Petroleum Zeitschrift*, 11, 1 (1930); also Edmund Graefe and Rudolf Schreiter, *Asphalt und Strassenbau*, 11, 3 (1930).

5 [p. 60] "Standard Definitions of Terms Relating to Materials for Roads and Pavements" (D 8–33), American Soc. for Testing Materials, Standards 1936, II, 1116.

CHAPTER III

1 [p. 66] "The Physical Condition of Machine Greases, Oil Solutions of Lime Soaps and Heavy Mineral Oils," by David Holde, *Z. angew. Chem.*, 31, 2138 (1908); "Bereiding en Constitutie van Asphalt," by F. J. Nellensteyn, Dissertation Delft (1923); *J. Inst. Petrol. Tech.*, 10, 311, (1924); "Constitution of Asphalt, by F. J. Nellensteyn, *Chem. Weekblad*, 21, 42 (1924); *J. Inst. Petroleum Tech.*, 10, 311 (1924); "New Asphalt Theories," by F. J. Nellensteyn, Intern. Congress Testing Materials, II, 684 (1927); "Constitution of Asphalt Micella," by F. J. Nellensteyn, *Chem. Weekblad*, 24, 414 (1927); "Colloidal Chemistry," by Jerome Alexander, Vol. III, pp. 491, 503 and 523, Sections entitled "Collidal Chemistry of Petroleum," by A. E. Dunstan, J. C. Morrell and Gustav Egloff, also Leo Gurwitsch, The Chemical Catalog Co., Inc., New York (1931).

2 [p. 70] "The Relation in Composition of the Different Forms of Natural Bitumens," by C. F. Mabery, *J. Am. Chem. Soc.*, 39, 2015 (1917); "Distillation of Pitches and Bitumens in a Cathodic Vacuum as a Means of Characterization," by A. Vila, *Compt. rend.*, 203, 1166 (1936).

3 [p. 70] "Chemical Examination of Asphaltic Material," by S. W. Parr, B. Mears and D. L. Weatherhead, *J. Ind. Eng. Chem.*, 1, 751 (1909); "The Lubricant and Asphaltic Hydrocarbons in Petroleum," by C. F. Mabery, *J. Ind. Eng. Chem.*, 45, 1233 (1923).

4 [p. 70] "A Chemical Investigation of the Asphalt in the Tar Sands of Northern Alberta," by V. K. Krieble and W. F. Seyer; *J. Am. Chem. Soc.*, 43, 1337 (1921).

5 [p. 70] "The Proximate Composition and Physical Structure of Trinidad Asphalt, with Special Reference to the Behavior of Mixtures of Bitumen and Fine Mineral Matter," by Clifford Richardson, *Proc. A.S.T.M.*, 6, 509 (1906); "Unique Geophysical Phenomenon, Trinidad Asphalt, Interesting from the Point of View of Dispersoid Chemistry," by Clifford Richardson, *J. Phys. Chem.*, 19, 241 (1915); "The Theory of the Perfect Sheet-Asphalt Surface," by Clifford Richardson, *J. Ind. Eng. Chem.*, 7, 463 (1915); "An Interesting Application of Colloidal Chemistry," by D. T. Pierce, *Met. Chem. Eng.*, 13, 408 (1915); "Colloids and Their Occurrence in Trinidad Lake Asphalt," by J. S. Miller, Jr., J. Eng. Club, Baltimore, Md., 6, 131 (1916); "Importance of the Relation of Solid Surfaces and Liquid Films in Some Types of Engineering Construction," by Clifford Richardson, Proc. Western Soc. Engineers, Chicago, Ill. (Nov. 20, 1916); *Sci. Amer. Supp.*, 83, No. 2152, 198 (1917); "The Colloidal State of Matter in its Relation to the Asphalt Industry," by Clifford Richardson, *Met. Chem. Eng.*, 17, 650 (1917); *J. Inst. Petrol. Tech.*, 8, 79 (1922); "Some Observations on the Colloidal Character of Asphalts," by R. E. Kirk and L. H. Reyerson, *J. Phys. Chem.*, 29, 865 (1925); "Colloidal Structure of Asphalts," by R. Wilhelmi, *Erdöl und Teer*, 8, 320, 335, 352, 368, 400, 416 and 431 (1932).

6 [p. 71] "Note on Petroleum," by W. Ramsay, *J. Soc. Chem. Ind.*, 42, 287T (1923).

7 [p. 71] "The Association of Vanadium with Petroleum and Asphalt," by R. M. Bird and W. S. Calcott, Bull. Philos. Soc. Univ. of Virginia, 1, (No. 18) (1914); "Petroleum Ash," by W. H. Thomas, *J. Inst. Petrol. Tech.*, 10, 216 (1924); "Metallic Constituents of Crude Petroleum," by W. B. Shirey, *J. Ind. Eng. Chem.*, 23, 1151 (1931).

8 [p. 71] "Vanadium Minerals in Argentina," by G. Fester and F. Bertuzzi, *Z. angew. Chem.*, 38, 363 (1925).

9 [p. 71] "The Vanadiferous Asphaltites of Central Peru," by J. G. Baragwanath, *Eng. Mining J.*, 111, 778 (1921); "Petroleum, Asphalt and Vanadium," by G. A. Fester and Jose Cruellas, Rev. facultad quim. ind. agr. (Univ. nacl. litoral), 4, 186 (1936).

10 [p. 71] "Metallic Constituents in Crude Petroleum," by W. B. Shirey, *Ind. Eng. Chem.*, 23, 1151 (1931).

11 [p. 71] "Uranium Bearing Asphaltite Sediments of Utah," by F. L. Hess, *Eng. Mining J.*, 114, 272 (1922).

12 [p. 71] **U. S. Pat.** 1772960 of Aug. 12, 1930 to Alfred Oberle.

13 [p. 71] "Feststellung von Vanadin- und Molybdänvorkommen in Holzheimer Schiefer," by Karl Neubronner, *Chem. Ztg.*, 49, 409 (1925).

14 [p. 71] "Free Carbon in Coal Tar and Pitch," by R. Hodurek, *Mitt. Inst. Kohlenvergasung*, 1, 9, 19 and 28 (1919).

15 [p. 72] "Effect of Continued Heating on Asphalts," by A. W. Sikes and C. H. Corey, *J. Ind. Eng. Chem.*, 27, 192 (1935).

16 [p. 72] **U. S. Pats.** 11203, 11204 and 11205 of Jun. 27, 1854 to Abraham Gesner.

17 [p. 74] "Petroleum Analytical Methods," by S. P. Sadtler, 8th Intern. Cong. Applied Chem., 25, 729–733 (1912).

18 [p. 75] "Hydrogenation by the Bergius Process," *J. Soc. Chem. Ind.*, 44, 471 (1925); **U. S. Pats.** 1251954 of Jan. 1, 1918 to Friedrich Bergius and J. Billwiller; 1342790 of Jun. 8, 1920 to Friedrich Bergius; 1344671 of Jun. 29, 1920 to Friedrich Bergius; 1391664 of Sep. 27, 1921 to Friedrich Bergius; 1592772 of Jul. 13, 1926 to Friedrich Bergius; 1607939 of Nov. 23, 1926 to Friedrich Bergius; **Brit. Pats.** of 1914 (Aug. 1), 18232 to Friedrich Bergius; of 1915 (Mar. 31), 5021 to Friedrich Bergius; 148436 of Jul. 10, 1920 to Friedrich Bergius; **Ger. Pats.** 303893 of Apr. 3, 1914 to Freidrich Bergius and J. Billwiller; 336334 of Dec. 24, 1918 to R. Tern.

19 [p. 75] **U. S. Pat.** 2029895 of Feb. 4, 1936 to W. Rittmeister; **Brit. Pat.** 247586 of Feb. 11, 1926 to I. G. Farbenindustrie A.-G.

20 [p. 75] **Can. Pat.** 318095 of Dec. 15, 1931 to N. V. de Bataafsche Petroleum Maatschappij.

21 [p. 76] "Revolution in Synthetic Fuel," New York "Sunday Times," May 11, 1930.

22 [p. 76] "Hydrogenation of Petroleum," by R. T. Haslam and R. P. Russell, *J. Ind. Eng. Chem.*, **22**, 1030 (1930).

23 [p. 76] "Recent Progress in Hydrogenation of Petroleum," by P. J. Byrne, Jr., E. J. Gohr and R. T. Haslam, *J. Ind. Eng. Chem.*, **24**, 1129 (1932).

24 [p. 77] **Brit. Pat.** 296431 of Aug. 8, 1928 to I. G. Farbenindustrie A.-G.

25 [p. 77] **French Pat.** 744220 of Oct. 17, 1932 to International Hydrogenation Patents Co.

26 [p. 77] **U. S. Pat.** 2065201 of Dec. 22, 1936 to Antoni Szayna.

27 [p. 77] "Hydrogenation of Low-Temperature Tar Products," by G. T. Morgan and J. T. Veryard, *J. Soc. Chem. Ind.*, **51**, 80T (1932); "Die Hydrierung von Steinkohlenschwelteer und Teererzeugnissen," by Anon., *Teer u. Bitumen*, **18**, 224 (1935).

28 [p. 77] "Treatment of Tar," by J. G. King and M. A. Matthews, *J. Inst. Fuel*, **6**, 33 (1932).

29 [p. 77] "On the Hydrogenation of Bitumen from the Bituminous Sands of Alberta," by E. H. Boomer and A. W. Saddington, *Can. J. Research* (Ottawa), **2**, 376 (1930); "Hydrogenation of Alberta Bitumen," by T. E. Warren, Bull. 725, Canadian Dept. Mines (1930–1); "Report of Experimental Work on the Hydrogenation of Canadian Coal, Coal Tar and Bitumen for the Production of Motor Fuel," by T. E. Warren and A. R. Williams, Dept. Mines, Ottawa, Canada, J. O. Patenaude, Ottawa (1933); **French Pat.** 523733 of Sep. 8, 1920 to Canadian-American Finance and Trading Co., Ltd.

30 [p. 77] "Neuere Beobachtungen aus der Destillations- und Hydrierungspraxis von Teeren und Oelen," by Fritz Frank, *Petroleum Z.*, **19**, 907 (1923); **Can. Pat.** 235924 of Nov. 27, 1923 to Meilach Melamid; **Brit. Pats.** 171367 of Oct. 11, 1921 to Meilach Melamid; 180625 of Nov. 10, 1921 to Meilach Melamid; 193922 of Nov. 10, 1921 to Meilach Melamid; **Ger. Pat.** 336334 of Dec. 24 ,1918 to R. Tern.

31 [p. 78] "Bestimmung die Carbüre in Mineralöldestillaten, sowie in Leucht- und Schmieröldestillaten," by Lazar Edeleanu, *Chem. Ztg.*, **38**, 391 (1914); "The Edeleanu Oil-Refining Process," by Anon., *Petroleum Times*, **10**, 916 (1923).

32 [p. 78] "The Edeleanu Process for Refining Petroleum," by R. L. Brandt, *J. Ind. Eng. Chem.*, **22**, 218 (1930).

33 [p. 78] **Hungarian Pat.** 103004 of Nov. 3, 1928 to László Bolgár.

34 [p. 78] "Action of Nitric Acid on Asphalt and Cellulose," by R. A. Fessenden, *Chem. Zentr.*, **21**, 686 (1892); **Austrian Pat.** 74127 of Dec. 15, 1916 to A. S. Flexer.

35 [p. 78] **Hungarian Pat.** 104882 of Feb. 16, 1928 to László Bolgár.

36 [p. 79] **Ger. Pats.** 486022 of Apr. 3, 1927 to A. M. Nastukoff; 509305 of Apr. 3, 1927 to A. M. Nastukoff.

37 [p. 79] **U. S. Pat.** 1201601 of Oct. 17, 1916 to Meilich Melamid; **Ger. Pats.** 276765 of Aug. 19, 1913 to Meilich Melamid; 332112 of Apr. 5, 1917 to Meilich Melamid; **French Pat.** 633542 of Apr. 28, 1927 to F. C. Bunge.

38 [p. 80] "Production of Bitumens and Asphaltums by the Action of Sulphur Chloride," by G. A. Leroy, *J. Soc. Chem. Ind.*, **26**, 754 (1907); "Observations on the Action of Sulphur Monochloride on Bituminous and Tarry Substances and Hydrocarbon Oils," by J. V. Meigs, *J. Ind. Eng. Chem.*, **9**, 655 (1917); **Brit. Pat.** 148003 of Jul. 29, 1920 to G. A. Henderson; **Ger. Pat.** 410012 of Mar. 15, 1923 to Farbwerke vorm. Meister Lucius & Brüning and G. Balle.

39 [p. 80] "Die Einwirkung von Selen auf hochsiedende Mineralöle," by Karl Graefe, *Z. angew. Chem.*, **34**, 509 (1921).

40 [p. 80] "The Formation of Asphalt in Petroleum," by R. Zaloziecki and C. Zelinski, 8th Intern. Cong. Applied Chem., **10**, 335 (1912); **Ger. Pat.** 332112 of Apr. 5, 1917 to Meilach Melamid and L. Grötzinger.

41 [p. 80] "The Action of Iodine on Hydrocarbons," by F. J. Nellensteyn, *Chem. Weekblad*, **21**, 102 (1924).

42 [p. 80] **Can. Pat.** 297208 of Feb. 4, 1930 to E. W. Hultman.

43 [p. 80] "Die Jodzahl von Natur- und Erdölasphalten," by Hans Pöll, Petroleum, **27**, 817 (1931); *Erdöl u. Teer*, **7**, 350 (1931).

44 [p. 80] "Chlorination of Natural Asphalts," by H. Loebell, *Chem. Rev. Fett- u. Harz-*

Ind., **18**, 165 (1911); **U. S. Pat.** 100876 of Mar. 15, 1870 to Alonzo Farrar; **Ger. Pat.** 406689 of Sep. 24, 1922 to Farbewerke vorm. Meister Lucius & Brüning.

45 [p. 80] **U. S. Pat.** 2059051 of Oct. 27, 1936 to J. W. Sparks; **Ger. Pat.** 406689 of Sep. 24, 1922 to Farbewerke vorm. Meister Lucius & Brüning.

46 [p. 80] **Ger. Pat.** 567100 of Mar. 9, 1930 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger A.-G.

47 [p. 80] **Ger. Pat.** 228497 of Nov. 25, 1909 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich.

48 [p. 80] **Brit. Pat.** 324774 of Dec. 21, 1928 to I. G. Farbenindustrie A.-G.

49 [p. 80] **Brit. Pat.** 379632 of Feb. 27, 1932 to Paul Lechler.

CHAPTER IV

1 [p. 91] "Conversion of Algae into Bitumen and Petroleum," by J. E. Hackford, *J. Inst. Petrol. Tech.*, **18**, 74 (1932).

2 [p. 92] "Formation of Asphaltic Bitumen from Proteins," by F. J. Nellensteyn and Dirk Thöenes, *Chem. Weekblad*, **30**, 359 (1933).

3 [p. 92] "Bituminous Substances," by P. E. Spielmann, Ernest Benn, Ltd., London, pp. 17–18 (1925); "Bitumen in Meteorites," by P. E. Spielmann, Nature, **114**, 276 (1924); "Origin of Asphaltic Bitumen," by Dirk Thöenes, Dissertation Tech. Hoogeschool, Delft (1936).

4 [p. 93] "The Relations of the Chemical Composition of Petroleum to its Genesis and Geologic Occurrence," by C. F. Mabery, *Econ. Geol.*, **11**, 511 (1916); "The Genesis of Petroleum as Revealed by its Nitrogen Constituents," by C. F. Mabery, *J. Am. Chem. Soc.*, **41**, 1690 (1919).

5 [p. 93] "The Origin of Petroleum and Asphalt," by Clifford Richardson, *J. Ind. Eng. Chem.*, **8**, 3 and 493 (1916); "The Nature and Origin of Asphalt," by Clifford Richardson, *Met. Chem. Eng.*, **16**, 25–27, (1917); St. Paul (Minn.) *Eng. Soc.* (1917); also *J. Soc. Chem. Ind.*, **37**, 59A (1918).

6 [p. 94] "The Relations in Composition of the Different Forms of Natural Bitumens," by C. F. Mabery, *J. Am. Chem. Soc.*, **39**, 2015 (1917); "Origin of Asphalt and Coal," by Julius Marcusson, *Chem. Zeit.*, **44**, 43 (1920).

7 [p. 95] "Petroleum and Coal," by Julius Marcusson, *Chem. Ztg.*, **42**, 437 (1918); "Asphalt and Coal," by Julius Marcusson, *Z. angew. Chem.*, **32**, 113, (1919); *Chem. Abs.*, **13**, 2592 (1919); also Mitt. k. Material-prüfungsamt, **38**, 307 (1920).

8 [p. 96] "Peat: Its Use and Manufacture," by P. E. Björling and F. T. Gissling, Chas. Griffin & Co., Ltd., London (1907); "Commercial Peat: Its Use and Possibilities," by F. T. Gissling, Chas. Griffin & Co., Ltd., London (1909); "Peat Industry Reference Book," by F. T. Gissling, Chas. Griffin & Co., Ltd., London (1920).

9 [p. 96] "The Data of Geochemistry," by F. W. Clarke, Bull. No. 330, U. S. Geol. Survey, Wash., D. C. (1908); "Notes on Lignite, its Characteristics and Utilization," by S. M. Darling, Tech. Paper No. 178, Bureau of Mines, Wash., D. C. (1919).

10 [p. 96] "Cannel Coal in the United States," by G. H. Ashley, Bull. No. 659, *U. S. Geol. Survey*, Wash., D. C. (1918).

11 [p. 97] "Chemistry and Analysis of Asphaltum," by Julius Marcusson, *Chem.-Ztg.*, **38**, 813 and 822 (1914); "Relations in Composition of Different Forms of Natural Bitumens," by C. F. Mabery, *J. Am. Chem. Soc.*, **39**, 2015 (1917); "Nature of Coal," by J. E. Hackford, *Mining and Met.*, **163**, 35 (1920); "Significance of the Interpretation of the Chemical Analysis of Oil Seepages," by J. E. Hackford, *J. Inst. Petrol. Tech.*, **8**, 193 (1922).

CHAPTER V

1 [p. 98] "The Production of Asphaltum and Bituminous Rock in 1904," by E. O. Hovey (1905); "The Production of Asphalt, Related Bitumens and Bituminous Rock in 1908," by J. A. Taff (1909); "The Production of Asphalt, Related Bitumens and Bituminous Rock in

1909," by D. T. Day (1911); "The Production of Asphalt, Related Bitumens and Bituminous Rock in 1910," by D. T. Day (1911); "Asphalt, Related Bitumens and Bituminous Rock in 1915," by J. D. Northrop (1916); "Asphalt, Related Bitumens and Bituminous Rock in 1917," by J. D. Northrop (1918); "Asphalt and Allied Substances in 1918," by C. C. Osbon (1920); "Asphalt and Related Bitumens in 1919," by K. W. Cottrell (1921); "Asphalt and Related Bitumens in 1920," by K. W. Cottrell (1921); "Asphalt and Related Bitumens in 1921," by K. W. Cottrell (1922); "Asphalt and Related Bitumens in 1922," by K. W. Cottrell (1923); "Asphalt and Related Bitumens in 1923," by K. W. Cottrell (1924); all published by the Dept. of Interior, U. S. Geol. Survey, Wash., D. C.; "Asphalt and Related Bitumens in 1924," by K. W. Cottrell (1926); "Asphalt and Related Bitumens in 1925," by G. R. Hopkins and A. B. Coons (1926); "Asphalt and Related Bitumens in 1926," by G. R. Hopkins (1927); "Asphalt and Related Bitumens in 1927," by G. R. Hopkins and A. B. Coons (1928); "Asphalt and Related Bitumens in 1928," by A. H. Redfield (1930); "Asphalt and Related Bitumens in 1929," by A. H. Redfield (1931); "Asphalt and Related Bitumens in 1930," by A. H. Redfield (Dec. 21, 1931); "Asphalt and Related Bitumens in 1931," by A. H. Redfield (Feb. 2, 1933); "Asphalt and Related Bitumens (1932)," by A. H. Redfield (1933); "Asphalt and Related Bitumens (1933)," by A. H. Redfield (1934); "Asphalt and Related Bitumens (1934)," by A. H. Redfield (1935); "Asphalt and Related Bitumens (1935)," by A. H. Redfield (1935); all published by the Dept. of Commerce, Bureau of Mines, Wash, D. C. "Contributions to Economic Statistics of the Asphalt Industry," by W. Schmidt, *Petroleum*, 27, (Supp.), 3 and 4 (1931).

2 [p. 106] Report of U. S. Tariff Commission, Tariff Series No. 33 (1925), also from other sources; "Coke and Byproducts," by L. N. Plein, W. H. Young and H. L. Bennit, Chapter from Minerals Yearbook 1935, Part. III, pp. 649–690, Bureau of Mines, U. S. Dept. Interior, Wash., D. C. (1935).

3 [p. 106] "Coal Tar," by S. R. Church, *Chem. Met. Eng.*, 37, 35 (1930); "Fifteenth Census of the United States; Manufactures: 1929"; Volume II, p. 744, U. S. Department of Commerce, Wash., D. C. (1933).

4. [p. 107] Private communication from the Asphalt Institute, New York City.

5 [p. 107] "Mileage of Improved Roads Completed and Under Construction as of Jan. 1st, 1936," by Anon., American Highways, 15, No. 2 (Apr. 1936); 16, No. 2 (Apr. 1937).

6 [p. 109] "Fifteenth Census of the United States"; Manufactures: 1929; Volume II, pp. 1350 and 1352, U. S. Department of Commerce, Wash., D. C. (1933); Census of Manufactures 1935, "Roofing, Built-up and Roll; Asphalt Shingles; Roof Coatings Other than Paint," Industry Release No. 1638; Department of Commerce, Bureau of Census, Wash., D. C. (Feb. 4, 1937).

7 [p. 111] "Fifteenth Census of the United States"; Manufactures: 1929; Volume II, p. 322, U. S. Department of Commerce, Wash., D. C. (1933); Census of Manufactures 1935, "Asphalted Felt-Base Floor Covering; Linoleum," Industries Nos. 222a and 222b, Department of Commerce, Bureau of Census, Wash., D. C. (Dec. 30, 1936).

CHAPTER VI

1 [p. 113] "Shipping of Asphalt," by C. S. Brewster, The Atlantic Connecting Rod, 15, 1 (1922).

2 [p. 114] **U. S. Pat.** 1391969 of Sep. 27, 1921 to F. C. Overbury and C. E. Rahr.

3 [p. 114] **U. S. Pats.** 1565798 of Dec. 15, 1925 to E. R. Dillehay and A. R. Lukens; 1666730 of Apr. 17, 1928 to Fred Breeze, Jr.; 1916970 of Jul. 4, 1933 to P. S. Denning.

4 [p. 114] **Russian Pat.** 33625 of Dec. 31, 1933 to G. N. Milbert.

5 [p. 114] **U. S. Pat.** 1734189 of Nov. 5, 1929 to J. H. Young.

6 [p. 114] **U. S. Pats.** 2015290 of Sep. 24, 1935 to J. McC. Sanders; 2047385 of Jul. 14, 1936 to J. McC. Sanders; **Brit. Pat.** 381632 of Jul. 23, 1931 to J. McC. Sanders.

7 [p. 114] **U. S. Pat.** 408951 of Aug. 13, 1889 to C. S. Bushnell; **Can. Pat.** 247179 of Feb. 24, 1925 to Texas Co.

8 [p. 114] **U. S. Pat.** 1912142 of May 30, 1933 to C. W. Hunt and A. H. McRae.

9 [p. 115] "Gas-fired Immersion Heating for Asphalt and Bitumen," by L. G. A. Leonard, *Gas World*, 97, No. 2520, Ind. Gas Suppl., p. 106 (1932); **U. S. Pat.** 1915056 of Jun. 20, 1933 to J. H. Fichter.

10 [p. 115] **Ger. Pats.** 16322 of May 11, 1881 to Wilhelm Jeffke; 24489 of Oct. 21, 1882 to Richard Michelet and Louis Tescher; 381515 of Oct. 11, 1922 to J. T. M. Johnston; 442232 of Aug. 5, 1925 to E. Weichert and J. Beyler.

11 [p. 116] "The Asphalt Industry," by Clifford Richardson, Proc. Engineers' Club (Phila.), 24, 13 (1917); **U. S. Pat.** 1604988 of Nov. 2, 1926 to F. H. Gilchrist; **Can. Pat.** 260004 of Apr. 20, 1926 to F. H. Gilchrist.

12 [p. 116] **Brit. Pat.** 447789 of Feb. 7, 1935 to J. Molyneux, F. Williams, H. H. Dewhurst, F. Hoon and G. S. Molyneux.

13 [p. 116] **U. S. Pat.** 1975433 of Oct. 2, 1934 to E. O. Rhodes; **Brit. Pat.** 454132 of Mar. 3, 1936 to International Hydrogenation Patents Co., Ltd.

14 [p. 117] "An Oil Heating System for Industrial Processes," by Anon., *Chem. Met. Eng.*, 17, 717 (1917); 19, 733 (1918); "Taking All Factors into Consideration, Gas can be Proved Least Expensive as Well as Most Economical Fuel for Asphalt Melting," by C. H. Kallstedt, *Am. Gas Eng. J.*, 113, 345 (1920); "Fluid Heat Transmission for High Temperatures in Industrial Processes," by J. A. Reavell, *J. Soc. Chem., Ind.*, 45, 825 (1926); *Gas Age*, 58, 35 (1926); *Chem. App.*, 13, 115 and 154 (1926); **U. S. Pats.** 1252376 of Jan. 1, 1918 to W. C. Merrill; 1636271 of Jul. 19, 1927 to Joseph Zavertnik, Jr.

15 [p. 117] "Heating Asphalt by Diphenyl Vapor," by G. H. Montillon, K. L. Rohrbach and W. L. Badger, *J. Ind. Eng. Chem.*, 23, 763 (1931); "Heating Asphalt with Diphenyl Vapor," by W. L. McCabe, Engineering Research Bull. No. 23, Univ. Michigan, Ann Arbor, Mich. (1932).

16 [p. 117] **U. S. Pat.** 1874258 of Aug. 30, 1932 to H. H. Dow.

17 [p. 117] **U. S. Pats.** 1165367 of Dec. 21, 1915 to G. R. Wyman; 1528822 of Mar. 10, 1925 to F. H. Gilchrist; 1757707 of May 6, 1930 to H. A. Dreffein; **Can. Pats.** 163719 of Jul. 13, 1915 to C. S. Bird; 169982 of Jun. 6, 1916 to C. S. Bird; **Ger. Pat.** 295863 of Apr. 16, 1916 to Benno Schilde and Adolf Boleg.

18 [p. 117] **U. S. Pats.** 512348 of Jan. 9, 1894 to W. S. Wilkinson; 512494 of Jan. 9, 1894 to R. D. Upham; 580592 of Apr. 13, 1897 to A. F. L. Bell.

19 [p. 118] **U. S. Pat.** 1182601 of May 9, 1916 to M. J. Trumble.

20 [p. 118] **Ger. Pat.** 110302 of Mar. 14, 1900 to Vereinigte Electricitäts A.-G.

21 [p. 118] **U. S. Pat.** 1163593 of Dec. 7, 1915 to C. N. Forrest; **Can. Pat.** 171345 of Aug. 15, 1916 to Barber Asphalt Paving Co.; **Ger. Pat.** 294046 of Dec. 3, 1915 to Barber Asphalt Paving Co.; **French Pat.** 480271 of Jul. 12, 1916 to Barber Asphalt Paving Co.

22 [p. 118] **U. S. Pats.** 1247937 of Nov. 27, 1917 to H. A. Cumfer; 1288631 of Dec. 24, 1918 to O. D. McFarland; 1349663 of Aug. 17, 1920 to H. A. Cumfer; 1389768 of Sep. 6, 1921 to O. D. McFarland; 1481255 of Jan. 22, 1924 to H. A. Cumfer; **Can. Pats.** 218671 of May 16, 1922 to Guyton & Cumfer Mfg. Co.; 232750 of Jul. 17, 1923 to Guyton & Cumfer Mfg. Co.; 251259 of Jun. 30, 1925 to Guyton & Cumfer Mfg. Co.

23 [p. 118] **Can. Pat.** 364315 of Feb. 23, 1937 to Philip Carey Mfg. Co.; **Ger. Pat.** 132101 of Mar. 1, 1901 to Anton Pieper.

24 [p. 119] **Ger. Pat.** 276196 of Feb. 14, 1914 to Carl Fohr and E. Kleinschmidt.

25 [p. 119] **U. S. Pat.** 551294 of Dec. 10, 1895 to Clifford Richardson.

26 [p. 120] **U. S. Pats.** 757387 of Apr. 12, 1904 to H. W. Ash; 779198 of Jan. 3, 1905 to H. W. Ash; 821323 of May 22, 1906 to Henry Wurtz; **Can. Pats.** 202622 of Aug. 10, 1920 to M. J. Gavin and C. P. Bowie; 222951 of Aug. 22, 1922 to J. H. Ginet; 230423 of Apr. 17, 1923 to Draper Mfg. Co.

27 [p. 120] **U. S. Pat.** 15506 of Aug. 12, 1856 to Luther Atwood and William Atwood.

28 [p. 120] **Can. Pat.** 147444 of Apr. 22, 1913 to J. H. Russell.

29 [p. 120] **U. S. Pat.** 1884618 of Oct. 25, 1932 to J. S. Downard.

30 [p. 121] **U. S. Pats.** 442441 of Dec. 9, 1890 to J. B. Jardine; 498957 of Jun. 6, 1893 to J. B. Jardine.

31 [p. 122] **U. S. Pat.** 498957 of Jun. 6, 1893 to J. B. Jardine.

32 [p. 122] **U. S. Pat.** 580592 of Apr. 13, 1897 to A. F. L. Bell.

33 [p. 122] "Refining Methods Used by Tar Springs Asphalt Co., Tar Springs, Okla." *Mines and Minerals* (Mar. 1903); "Asphalt Mining and Refining in Oklahoma," by W. R. Crane, *Eng. Mining J.*, (Dec. 17, 1903); "Preliminary Report on the Bituminous Sands of Northern Alberta," by S. C. Ells, Dept. Mines, Ottawa, Can. (1914); "Hot Water Separation of Bitumen from Alberta Bituminous Sand," by K. A. Clark and D. S. Pasternack, *J. Ind. Eng. Chem.*, **24**, 1410 (1932); **U. S. Pats.** 505416 of Sept. 19, 1893 to A. F. L. Bell; 507885 of Oct. 31, 1893 to A. S. Cooper; 549399 of Nov. 5, 1895 to H. L. Sellers and Thomas Conyngton; 655416 of Aug. 7, 1900 to Jacob Philippi; 722500 of Mar. 10, 1903 to J. S. Downard and B. A. Roloson; 918628 and 918629 of Apr. 20, 1909 to G. M. Willis; 1190633 of Jul. 11, 1916 to C. L. Cook and J. R. Price; 1514162 of Nov. 4, 1924 to A. F. Kelsey; 1520752 of Dec. 30, 1924 to William Horwitz; **Can. Pats.** 194319 of Dec. 2, 1919 to C. L. Cook and J. R. Price; 234961 of Oct. 16, 1923 to Louis Simpson; 235114 of Oct. 23, 1923 to Louis Simpson; 237770 of Feb. 12, 1924 to J. D. Tait; 245317 of Dec. 16, 1924 to William Georgeson; 326747 of Oct. 11, 1932 to International Bitumen Co., Ltd.; **Ger. Pat.** 332100 of Apr. 1, 1919 to William Horwitz.

34 [p. 122] **French Pat.** 637619 of Nov. 13, 1926 to Paul Vérola.

35 [p. 122] "The Extraction of Bitumens from Mineral Aggregate," by M. R. Walczak and H. I. Rice, *J. Ind. Eng. Chem.*, **12**, 738 (1920).

36 [p. 122] **U. S. Pat.** 1253386 of Jan. 15, 1918 to I. S. Joseph.

37 [p. 123] "Changes Due to Weathering of Derna-Tataros Sand Asphalt," by J. Jachzel and G. Sachs, *Petroleum*, **30**, 1 (1934).

38 [p. 124] **U. S. Pat.** 597892 of Jan. 25, 1898 to W. S. Wilkinson.

39 [p. 124] "The Separation of Adherent Oil or Bitumen from Rock," by Ernest Fyleman, *J. Soc. Chem. Ind.*, **41**, 14T (1922); "Proposed Method for Mining Oil Sands," by M. A. Newman; *Petroleum Times*, **8**, 284 (1922); "The Bituminous Sands of Northern Alberta," by K. A. Clark, *3rd Ann. Report Sci. Ind. Research Council of Alberta*, **42** (1922); **U. S. Pat.** 1820917 of Sep. 1, 1931 to C. T. Langford and A. J. Teplitz; **Can. Pats.** 203676 of Sep. 7, 1920 to Ernest Fyleman; 234272 of Sep. 18, 1923 to J. M. McClave; 289058 of Apr. 23, 1929 to Governors of the Univ. of Alberta; **Brit. Pat.** 163519 of Mar. 3, 1920 to Ernest Fyleman; **French Pat.** 531583 of Mar. 2, 1921 to Ernest Fyleman.

40 [p. 124] J. M. McClave, Western Research Laboratories, Denver, Col. (1923); **U. S. Pat.** 1594695 of Aug. 3, 1926 to J. M. McClave.

41 [p. 124] "The Extraction of Bitumens from Mineral Aggregates," by M. R. Walczak and H. I. Rice, *J. Ind. Eng. Chem.*, **12**, 738 (1920); **U. S. Pat.** 1820917 of Sep. 1, 1931 to C. T. Langford and A. J. Teplitz; **Can. Pat.** 296405 of Jan. 7, 1930 to Continental Oil Co.

42 [p. 124] "Separation of Bitumen from Asphaltic Rocks of Ragusa by Treatment with Aqueous Saline Solutions," by G. Roberti, *Annali Chim. Appl.*, **21**, 269 (1931).

43 [p. 124] K. A. Clark, The Press Bulletin, Univ. of Alberta, Edmonton, Alberta, **9** (May, 1924); *Can. Mining Met. Bull*, **212**, 1385 (1930); **U. S. Pat.** 1791797 of Feb. 10, 1931 to K. A. Clark.

44 [p. 124] **U. S. Pat.** 1791797 of Feb. 10, 1931 to K. A. Clark.

45 [p. 124] **U. S. Pat.** 1822271 of Sep. 8, 1931 to G. W. Coggeshall.

46 [p. 125] "Use of Solvents for Dewaxing Paraffin-Base Crudes," Reports of Investigation of Bureau of Mines No. 2822, Wash., D. C. (Jul. 1927); "Gewinnung fester Bitumina durch Extraktion," by E. J. Fischer, *Teer u. Bitumen*, **28**, 341 (1930).

47 [p. 125] **U. S. Pat.** 617226 of Jan. 3, 1899 to A. S. Cooper; **French Pat.** 618647 of Jul. 7, 1926 to Soc. Internationale des Combustibles Liquides.

48 [p. 125] "Fractionation of Petroleum Residues and of Heavy Petroleum by Compressed Natural Gas," by S. von Pilat, *Oel und Kohle*, **11**, 655 (1935); **U. S. Pats.** 1944491 of Jan. 23, 1934 to U. B. Bray; 1949989 of Mar. 6, 1934 to U. B. Bray; 1968392 of Jul. 31, 1934 to R. E. Haylett; 1978361 of Oct. 23, 1934 to G. A. Beiswenger; 1988711 and 1988712 of Jan. 22, 1935 to U. B. Bray and C. E. Swift; 1988714 and 1988715 of Jan. 22, 1935 to U. B. Bray; 2002004 of May 21, 1935 to E. W. Gard; 2010007 and 2010008 of Aug. 6, 1935 to U. B. Bray; 2029290 of Feb. 4, 1936 to U. B. Bray and L. B. Beckwith; 2031204 of Feb. 18, 1936

to U. B. Bray; 2031234 of Feb. 18, 1936 to C. E. Swift; 2040239 of May 12, 1936 to U. B. Bray and C. E. Swift; 2041275, 2041276, 2041277 and 2041279 of May 19, 1936 to U. B. Bray; 2041278 of May 19, 1936 to B. G. Aldridge and Basil Hopper; 2049000 of Jul. 28, 1936 to R. B. Day; 2049046 of Jul. 28, 1936 to U. B. Bray; 2055135 of Sep. 22, 1936 to Mathias Pier and August Eisenhut; **Can. Pat.** 348720 of Mar. 5, 1935 to Union Oil Co.; 352059 of Jul. 30, 1935 to Union Oil Co.; 359103 and 359104 of Jul. 14, 1936 to Texaco Development Corp.; 360397 of Sep. 8, 1936 to Standard Oil Co.; **Ger. Pats.** 362458 of Nov. 13, 1921 to "Metan" Spolka; 615455 of Jul. 9, 1932 to I. G. Farbenindustrie A.-G.; Appl. J-45097 of Aug. 12, 1932 to I. G. Farbenindustrie A.-G.; Appl. J-45673 of Nov. 4, 1932 to I. G. Farbenindustrie A.-G.; Appl. J-50131 of Jul. 11, 1935 to I. G. Farbenindustrie A.-G.; 630538 of Mar. 16, 1934 to Standard Oil Development Co.; **French Pat.** 800144 of Jun. 27, 1936 to Standard Oil Development Co.

49 [p. 125] "Gewinnung fester Bitumina durch Extraktion," by E. J. Fischer, *Teer u. Bitumen*, **28**, 341 (1930); **U. S. Pats.** 452764 of May 19, 1891 to Frederick Salathé; 581546 of Apr. 27, 1897 to H. A. Frasch; 617712 of Jan. 17, 1899 to A. F. L. Bell; 655430 of Aug. 7, 1900 to A. F. L. Bell; 1060010 of Apr. 29, 1913 to S. R. Murray and G. E. McDermand; 1722872 of Jul. 30, 1929 to Anton Weindel; 1952647 of Mar. 27, 1934 to A. B. Alvey, G. H. Alvey and E. A. Young; **Can. Pats.** 197465 of Feb. 24, 1920 to Frank Navin; 207590 of Jan. 18, 1921 to Jesse Coogan; **Ger. Pats.** 15775 of Dec. 31, 1880 to J. Jossinet; 101373 of Jul. 1, 1897 to Edgar von Bogen; 185690 of Oct. 17, 1903 to David Holde; 339988 of Oct. 15, 1918 to Ludwig Landsberg; 340074 of Oct. 15, 1918 to Ludwig Landsberg; 410420 of Feb. 20, 1924 to A. Wohl, A. Prill and A. Goldschmidt; 430438 of Sep. 5, 1923 to Z. M. Stinnes; 433455 of Sep. 5, 1923 to Z. M. Stinnes; 436242 of Sep. 10, 1925 to W. Demann; 477499 of Nov. 20, 1923 to Z. M. Stinnes; 500280 of Mar. 27, 1926 to Prodorite, S. A.; **French Pat.** 529244 of Aug. 2, 1920 to Simon Carves.

50 [p. 125] **U. S. Pat.** 1707759 of Apr. 2, 1929 to W. H. Hampton; **Brit. Pat.** 365046 of Oct. 8, 1930 to I. G. Farbenindustrie A.-G.; **Ger. Pat.** 591729 of May 27, 1930 to I. G. Farbenindustrie A.-G.

51 [p. 125] **Ger. Pats.** 412423 of Nov. 25, 1919 to Shale Reduction Machinery Corp.; 432851 of Feb 1, 1924 to Fabrik Braunkohlen-Produkte A.-G.; Appl. K-129517 of Mar. 17 1933 to Martha Kabbert.

52 [p. 125] **U. S. Pat.** 452764 of May 19, 1891 to Frederick Salathé; **Brit. Pat.** 409278 of Oct. 20, 1932 to J. Y. Johnson; **Ger. Pats.** 368331 of Oct. 29, 1919 to Deutsche Erdöl A.-G.; 380331 of Sep. 26, 1922 to Carl Engler; 399280 of Mar. 29, 1923 to R. J. Lacau; 408030 of Apr. 6, 1924 to Z. M. Stinnes; 453175 of Dec. 28, 1923 to Z. M. Stinnes; 486475 of Jun. 16, 1925 to Z. M. Stinnes; **French Pat.** 590748 of Feb. 16, 1925 to R. J. Lacau.

53 [p. 125] **Ger. Pats.** 305349 of Aug. 9, 1916 to A. Riebeck'sche Montanwerke A.-G.; 341763 of May 15, 1919 to A. Riebeck'sche Montanwerke A.-G.; 352902 of May 15, 1919 to A. Riebeck'sche Montanwerke A.-G.

54 [p. 125] **Ger. Pat.** 432744 of Aug. 19, 1923 to A. Riebeck'sche Montanwerke A.G.

55 [p. 125] **Brit. Pat.** 129992 of Jul. 16, 1919 to H. D. Ryan; **Ger. Pat.** 412423 of Nov. 25, 1919 to Shale Reduction Machinery Corp.

56 [p. 125] **Ger. Pat.** 204256 of Sep. 21, 1906 to Hippolyt Köhler.

57 [p. 125] **Ger. Pat.** 411540 of May 7, 1922 to Ernst Berl.

58 [p. 125] "Nitrobenzene as a Selective Solvent for Use in Making Motor Oils of High-Viscosity Index," by S. W. Ferris and W. F. Houghton, *Oil and Gas J.*, **31**, No. 26, 65 (1932); "Petroleum Refining by Means of Selective Solvents," by S. W. Ferris, *J. Franklin Inst.*, **217**, 591 (1934).

59 [p. 125] **French Pat.** 730431 of Jan. 22, 1932 to Atlantic Refining Co.

60 [p. 125] "Petroleum Refining by Means of Selective Solvents," by S. W. Ferris, *J. Franklin Inst.*, **217**, 591 (1934); **U. S. Pat.** 2010423 of Aug. 6, 1935 to A. A. Wells; **Can. Pats.** 348889 of Mar. 12, 1935 to Standard Oil Development Co.; 361747 of Nov. 10, 1936 to Friedrich Uhde; **Ger. Pat.** 213507 of Jul. 31, 1908 to Chemische Fabrik Lindenhof, C. Weyl & Co.; **Brit. Pat.** 362580 of Oct. 6, 1930 to Burmah Oil Co., Ltd.; **Ger. Pat.** 245666 of Nov. 20, 1923 to Z. M. Stinnes and Anton Weindel.

61 [p. 125] **Brit. Pat.** 383801 of Jan. 22, 1932 to Atlantic Refining Co.

62 [p. 125] **U. S. Pat.** 1741555 of Dec. 31, 1929 to M. L. Chappell and G. J. Ziser; **Brit. Pat.** 362580 of Oct. 6, 1930 to Burmah Oil Co., Ltd.; **Ger. Pat.** 395597 of Jan. 6, 1923 to Robert Kattwinkel.

63 [p. 125] **U. S. Pat.** 452764 of May 19, 1891 to Frederick Salathé; **Ger. Pat.** 374929 of Nov. 16, 1918 to M. A. Rakusin.

64 [p. 125] **U. S. Pat.** 1836020 of Dec. 15, 1931 to Helmuth Freytag; **Ger. Pats.** 232657 of Mar. 6, 1910 to A. Riebeck'sche Montanwerke, A.-G.; 315554 of Jun. 15, 1918 to Röchling'sche Eisen- und Stahlwerke, G.m.b.H.; 436444 of Jul. 22, 1922 to Z. M. Stinnes; 437048 of Oct. 18, 1923 to Z. M. Stinnes; 437410 of Dec. 11, 1924 to Z. M. Stinnes; 451958 of Mar. 21, 1926 to Z. M. Stinnes; 517279 of Dec. 8, 1926 to I. G. Farbenindustrie A.-G.; 526671 of Aug. 19, 1926 to A. Riebeck'sche Montanwerke A.-G.; 574277 of Apr. 11, 1933 to A. Riebeck'sche Montanwerke A.-G.; **French Pat.** 597919 of Aug. 13, 1924 to A. Mailhe.

65 [p. 125] "New Method for Asphalt Recovery by the Refining of Lubricants with Selective Extraction," by R. Fussteig, *Teer u. Bitumen*, 33, 429 (1935); **U. S. Pats.** 1813327 of Jul. 7, 1931 to B. L. Souther and W. A. Gruse; 1825762 of Oct. 6, 1931 to René de M. Taveau; 1835600 of Dec. 8, 1931 to J. H. James; 2035112 of Mar. 24, 1936 to J. C. Bird; 2062355 of Dec. 1, 1936 to Carleton Ellis; **Can. Pat.** 347756 of Jan. 22, 1935 to Standard Oil Development Co.; 353486 of Oct. 8, 1935 to Standard Oil Development Co.; 362067 of Nov. 24, 1936 to Standard Oil Development Co.; **Brit. Pat.** 443287 of Feb. 24, 1936 to Kornel Kell and W. H. Schmitz; **Ger. Pats.** 124980 of Dec. 28, 1900 to Carl Daeschner; 176468 of Jul. 12, 1904 to Bernard Diamand; 385153 of Jan. 18, 1921 to Wilhelm Gintl and Karl Müller; 439005 of Jul. 22, 1922 to Siemens & Halske A.-G.; 532212 of May 12, 1927 to A. Riebeck'sche Montanwerke A.-G.; 549451 of Nov. 21, 1930 to Werschen-Weiszenfelser Braunkohlen A.-G.

66 [p. 125] **U. S. Pat.** 1905423 of Apr. 25, 1933 to E. O. Rhodes and F. D. Hager; **Can. Pat.** 336568 of Oct. 24, 1933 to American Tar Products Co., Inc.; **Ger. Pats.** 166452 of Sep. 24, 1904 to Ludwig Landsberg; 360745 of Sep. 3, 1919 to Ernst Erdmann; 372330 of Jul. 21, 1920 to Julius Pintsch A.-G.; 388467 of Apr. 19, 1922 to Edwin Benesch; 439006 of Nov. 8, 1923 to Siemens & Halske A.-G.; 640522 of Dec. 24, 1930 to W. R. Roederer; **Austrian Pat.** 90279 of Apr. 15, 1922 to Holzverkohlungs-Industrie A.-G.

67 [p. 125] **Ger. Pats.** 325165 of Jan. 25, 1919 to A. Riebeck'sche Montanwerke A.-G.; 389896 of Aug. 22, 1920 to Holzverkohlungs-Industrie A.-G.

68 [p. 125] **Ger. Pat.** 389393 of Aug. 22, 1920 to Holzverkohlungs-Industrie A.-G.; **Austrian Pat.** 90279 of Apr. 15, 1922 to Holzverkohlungs-Industrie A.-G.

69 [p. 125] **U. S. Pat.** 2010423 of Aug. 6, 1935 to A. A. Wells; 2000690 of May 7, 1935 to L. A. Clarke; **Ger. Pat.** 352521 of May 4, 1920 to Plauson's Forschungsinstitut, G.m.b.H.

70 [p. 125] **Ger. Pat.** 352917 of Feb. 18, 1921 to Deutsche Erdöl A.-G.

71 [p. 125] **Ger. Pats.** 191839 of Oct. 13, 1906 to Curt Koettnitz; 388467 of Apr. 19, 1922 to Edwin Benesch.

72 [p. 125] **U. S. Pats.** 911553 of Feb. 2, 1909 to Lazar Edeleanu; 1989045 of Jan. 22, 1935 to D. R. Merrill; **Ger. Pat.** 437210 of Nov. 12, 1925 to Deutsche Erdöl A.-G.

73 [p. 125] **U. S. Pats.** 452764 of May 19, 1891 to Frederick Salathé; 581451 of Apr. 27, 1897 to A. F. L. Bell; **Ger. Pat.** 410420 of Feb. 20, 1924 to A. Wohl, A. Prill and A. Goldschmidt.

74 [p. 125] **U. S. Pat.** 1409590 of Mar. 14, 1922 to Frederick Salathé; **Ger. Pat.** 430010 of Nov. 8, 1925 to I. G. Farbenindustrie A.-G.

75 [p. 125] **Ger. Pat.** 388467 of Apr. 19, 1922 to Edwin Benesch.

76 [p. 125] **U. S. Pat.** 2024221 of Dec. 17, 1935 to L. M. Henderson.

77 [p. 126] **Ger. Pat.** 173616 of Jul. 26, 1904 to Bernard Diamand.

78 [p. 126] **French Pat.** 759691 of Feb. 7, 1934 to I. G. Farbenindustrie A.-G.

79 [p. 126] **Ger. Pat.** 305349 of Aug. 9, 1916 to A. Riebeck'sche Montanwerke A.-G.

80 [p. 126] **Ger. Pat.** 325165 of Jan. 25, 1919 to A. Riebeck'sche Montanwerke A.-G.

81 [p. 126] **U. S. Pat.** 2055428 of Sep. 22, 1936 to R. J. Dearborn.

82 [p. 126] **Brit. Pat.** 365205 of Jun. 4, 1930 to J. Y. Johnson.

83 [p. 126] **Can. Pat.** 320424 of Mar. 8, 1932 to Standard Oil Development Co.; **French Pat.** 785349 of Aug. 7, 1935 to Standard Oil Development Co.

84 [p. 126] **Brit. Pat.** 256933 of Jun. 15, 1926 to Herbert Wittek; **Ger. Pat.** 386708 of Dec. 2, 1921 to Eduard Jena.

85 [p. 126] "Liquid Propane—Use in Dewaxing, Deasphalting and Refining Heavy Oils," by R. E. Wilson, P. C. Keith, Jr., and R. E. Haylett, *J. Ind. Eng. Chem.*, **28**, 1065 (1936).

86 [p. 128] "Recovery of Waste Roofing Felt," by L. F. Hawley and Otto Kress, *Paper*, **27**, 18 (1920).

87 [p. 128] **U. S. Pat.** 1378637 of May 17, 1921 to M. P. Wall.

CHAPTER VII

1 [p. 130] "The Composition of the Solid Hydrocarbons of Petroleum, Paraffine and Ceresine," by Julius Marcusson, *Mitt. K. Materialprüfungsamt*, **33**, 415 (1916); *Chem. Zentr.*, **1**, 1285 (1916).

2 [p. 130] **Can. Pats.** 285711 and 285712 of Dec. 18, 1928 to I. G. Farbenindustrie A.-G.; 287712 of Mar. 5, 1929 to I. G. Farbenindustrie A.-G.

3 [p. 132] "A Treatise on Ozokerite," by E. B. Gosling, School of Mines Quarterly, Columbia University, **16**, 41 (1894).

4 [p. 132] "Mineral-wax Production in Poland," U. S. Bureau of Foreign and Domestic Commerce, p. 309, U. S. Commerce Report (Jul. 31, 1922).

5 [p. 133] "Ozokerit, seine Stellung zum Erdöl und seine Vorkommen im Kaukasus und Anderweitig," by A. F. v. Stahl, private communication (1928); *Petroleum*, **25**, 351 (1929).

6 [p. 134] "The Ozokerite Industry on the Island of Cheleken," *Petroleum World*, **14**, 136 (1917).

7 [p. 134] "Petroleum and Residual Bitumens in Leyte," by W. E. Pratt, *Philippines J. Sci.*, **10**, No. 4, Sec. A (Jul., 1915).

8 [p. 134] "The Ozokerite Deposits of Soldier Summit, Utah," by W. C. Higgins, *Salt Lake Min. Rev.*, **18**, 16 (1916); "Ozokerite in Utah," by L. O. Howard, *Mining Sci. Press*, **112**, 907 (1916); "Ozokerite in Central Utah," by H. M. Robinson, Bull. No. 641, *U. S. Geol. Survey*, Wash., D. C. (1916); "The Natural Hydrocarbons: Gilsonite, Elaterite, Wurtzilite, Grahamite, Ozokerite, and Others," by R. B. Ladoo, Reports of Investigations, Bureau of Mines, Wash., D. C. (1920).

9 [p. 135] "Ozokerite from the Thrall Oil Field," by E. P. Schoch, *J. Ind. Eng. Chem.*, **8**, 1095 (1916).

10 [p. 136] **Ger. Pats.** 99566 of Mar. 5, 1898 to E. Meyer; 101373 of Jul. 1, 1897 to E. v. Boyen; 166453 of Oct. 10, 1899 to Montanwachsfabrik, G.m.b.H.; 204256 of Sep. 21, 1906 to Hippolyt Köhler.

11 [p. 136] **Brit. Pats.** 288197 and 288258 of Apr. 4, 1927 to A. Riebeck'sche Montanwerke A.-G.; **Ger. Pat.** 341763 of May 15, 1919 to A. Riebeck'sche Montanwerke A.-G.

12 [p. 136] "Increasing the Yield of Montan Wax by Extracting Lignite under Pressure," by Franz Fischer and Wilhelm Schneider, *Ges. Abhandl. zur Kentnniss der Kohle*, **1**, 204 (1917), **2**, 57 (1917).

13 [p. 136] "Braunkohlenteer-Industrie," by Edmund Graefe, Verlag von Wilhelm Knapp, Halle, a.S., Germany (1908).

14 [p. 137] "Distillation of Bituminous Lignite in Vacuo," by Wilhelm Schneider and H. Tropsch, *Chem. Abs.*, **13**, 2995 (1919).

15 [p. 137] **Ger. Pat.** 260697 of Mar. 29, 1911 to Montanwachsfabrik, G.m.b.H.

16 [p. 134] **U. S. Pats.** 1834865 and 1834866 of Dec. 1, 1931 to Karl Behringer.

17 [p. 137] **Ger. Pat.** 256856 of Nov. 19, 1910 to C. F. Boehringer & Söhne.

18 [p. 138] **Ger. Pat.** 539391 of Mar. 3, 1925 to Montan, Inc.

CHAPTER VIII

1 [p. 140] "The Hydrocarbons of Utah," by Carlos Bardwell, *J. Ind. Eng. Chem.*, 5, 973 (1913).

2 [p. 140] "Oil and Asphalt Prospects in the Salt Lake Basin, Utah," p. 473, Bull. No. 260, *U. S. Geol. Survey*, Wash., D. C. (1905).

3 [p. 141] "The Modern Asphalt Pavement," by Clifford Richardson, p. 205, John Wiley & Sons, New York (1908).

4 [p. 143] "Nineteenth Annual Report of The U. S. Geological Survey," Part III, p. 368, Wash., D. C. (1899); "Native Asphalts in Oregon," by R. T. Hodge, *Bull. Am. Assoc. Petrol. Geologists*, 11, 395 (1927).

5 [p. 143] "The Modern Asphalt Pavement," by Clifford Richardson, p. 197, John Wiley & Sons, New York (1908).

6 [p. 144] "Petróleo en Chiapas," by E. M. Espinosa, *Bol. del Petróleo*, 2, 22 (1916).

7 [p. 145] "The Bitumens of Cuba," by Clifford Richardson.

8 [p. 146] "Production of Trinidad and Bermudez Lake Asphalts," by C. N. Forrest, *Roads and Streets*, 5, 25–30 and 46–47 (1915); "The Lake Asphalt Industry," by J. S. Miller, Jr., *Chem. Met. Eng.*, 22, 749 (1920); "Petroleum and Asphalt in Venezuela," by A. H. Redfield, *Eng. Mining J.*, 111, 354 and 393 (1921); "The Chemistry of Bituminous Highway Construction—Native Asphalts," by J. S. Miller, Jr., *J. Soc. Chem. Ind.*, 48, 782 (1929).

9 [p. 148] "Petroleum and Asphalt in Venezuela," by A. H. Redfield, *Eng. Min. J.*, 111, 354 and 393 (1921).

10 [p. 148] "The Modern Asphalt Pavement," by Clifford Richardson, p. 183, John Wiley & Sons, New York (1908).

11 [p. 148] "The Hydrocarbons of Utah," by Carlos Bardwell, *J. Ind. Eng. Chem.*, 5, 973 (1913); 6, 865 (1914).

12 [p. 150] "The Modern Asphalt Pavement," by Clifford Richardson, pp. 190–191, John Wiley & Sons, New York (1908).

13 [p. 151] "Die Asphaltvorkommen Russlands," by A. F. v. Stahl, *Asphalt u. Teerindustrie Zeitung*, 26, 617 and 644 (1926).

14 [p. 152] "Reports from the Consuls of the U. S.," 42, 228 (1893); "Turkish Bitumens," by Anon., *J. Roy. Soc. Arts*, 56, 829 (1908); "Oil and Asphalt in Palestine, Syria and Mesopotamia," by Felix Oswald, *Petroleum World*, 15, 459 (1918); "The Mineral Resources of Syria," by I. M. Toll, *Eng. Min. J.*, 112, 846 (1921).

15 [p. 153] "Asphalt from Great Asphaltic Lake in Okha (Sakhalin)," by S. S. Nametkin and S. N. Pavlova, Nephtyanoye Chozyaistvo, 1, 86 (1931).

16 [p. 153] "Investigation of Saghalein Petroleum and Asphalt," by Anon., *Petrol. Rev. and Mining News*, 9, No. 237, 230 (1903).

17 [p. 153] "Asphalt and Petroleum in the Philippines," by J. F. Boomer, *U. S. Commerce Report* No. 170, 358, Wash., D. C. (1915); "Rock Asphalt in Philippines," by J. F. Boomer, *U. S. Commerce Report* No. 193, 856, Wash., D. C. (1915); "Asphalt from the Philippines," by Anon., *Chamber of Commerce J.*, 34, 96 (1915); *Philippine J. Science*, 10A, 241 (1915); "Solid Bitumens of Leyte," by B. Nelson, *Philippine J. Science*, 22, 617 (1923); "Asphaltite in the Philippine Islands," by E. T. Hodge, *Philippine J. Science*, 37, 263, (1928).

CHAPTER IX

1 [p. 155] "Asphaltum Deposits of California," Mineral Resources of the U. S. 1883–4, pp. 938–948, *U. S. Geol. Survey*, Gov. Printing Office, Wash., D. C. (1884); "Asphaltum," by Clifford Richardson and E. W. Parker, Mineral Resources of the U. S. 1893, pp. 627–669, *U. S. Geol. Survey*, Gov. Printing Office, Wash., D. C. (1894); "The Genesis of Petroleum and Asphaltum in California," by A. S. Cooper, Bull. No. 16, California State Mining Bureau, Sacramento, Cal. (1899); "The Asphalt and Bituminous Rock Deposits of the U. S.," by G. H. Eldridge, 22nd Annual Report of the U. S. Geol. Survey, Part I, 1901, Gov. Printing Office, Wash., D. C. (1901).

2 [p. 155] "Occurrences of Petroleum, Natural Gas and Asphalt Rock in Western Ken-

tucky," by Edward Orton, *Geol. Survey of Kentucky* (1891); "The Asphalt and Bituminous Rock Deposits of the United States," by George H. Eldridge, 22nd Annual Report, *U. S. Geol. Survey*, Wash., D. C., Part I, p. 240 (1901); "Mining of Kentucky Rock Asphalt," by G. D. Steele, *Better Roads and Streets*, **5**, 15 (1915); "Economic Papers on Kentucky Geology," by W. R. Jillson, *Kentucky Geol. Survey*, Frankfort, Ky., pp. 39–57, Chap. II (1921); "Kentucky Rock Asphalt," by W. R. Jillson, *Pan-Amer. Geologist*, **41**, 251 (1924).

3 [p. 159] "Biennial Report of the State Geologist," Missouri Bureau of Geology and Mines, Rolla, Missouri (1919).

4 [p. 159] "Contributions to Economic Geology 1902," Bull. No. 213, *U. S. Geol. Survey*, Wash., D. C., p. 333 (1903).

5 [p. 164] "Asphalt and Petroleum in Oklahoma," by L. L. Hutchison, Bull. No. 2, *Okla. Geol. Survey*, Norman, Okla. (Mar., 1911); "Rock Asphalts in Oklahoma and their Use in Paving," by L. C. Snider, Circ. No. 5, *Okla. Geol. Survey*, Norman, Okla. (Apr., 1913); "The Occurrence of Asphalt in the State of Oklahoma," by C. N. Gould, *J. Royal Soc. Arts*, **63**, 132 (1915); "Native Road Materials and Highway Maintenance," by N. E. Wolfard, Circ. No. 20, *Okla. Geol. Survey*, Norman, Okla. (1929).

6 [p. 165] "Rock Asphalts of Oklahoma and Their Use in Paving," by L. C. Snider, *Petroleum*, **9**, 974 (1914).

7 [p. 167] "Composition of Blown Alberta Bitumen," by M. Katz, *Canadian J. Research*, **10**, 435 (1934).

8 [p. 167] "Accelerated Weathering Properties of Oklahoma Asphalts," by P. G. Shelley, Circ. No. 19, *Okla. Geol. Survey*, Norman, Okla. (Oct., 1929).

9 [p. 167] "Asphalt Deposits of Pike County, Arkansas," by C. W. Hayes, pp. 353–355, Bull. No. 213, *U. S. Geol. Survey*, Wash., D. C., (1913); "Asphalt Deposits and Oil Conditions in Southwestern Arkansas," by H. D. Miser and A. H. Purdue, Part II, p. 272, Bull. No. 691, *U. S. Geol. Survey*, Wash., D. C. (1918).

10 [p. 168] "Second Biennial Report on the Geology of Alabama," by M. Tuomey, p. 41, State of Alabama (1858); "Report on the Valley Regions of Alabama," by Henry McCalley, Part I, State of Alabama (1896); "Rock Asphalts of Alabama and their Use in Paving," by G. H. Clark, Spec. Report No. 13, *Geol. Survey of Alabama*, Univ. of Alabama (1925); "Summary of the Distribution and Occurrence of the Rock-Asphalt Deposits of Alabama," by W. B. Jones, Circ. No. 5, *Geol. Survey of Alabama*, University of Alabama (1928).

11 [p. 169] "Valuable Asphalt and Gypsum Deposits in Southern Louisiana," by W. D. Robinson, *Manufacturers' Record*, **71**, 64 (May 17, 1917).

12 [p. 169] "Uvalde Folio," by T. W. Vaughan, U. S. Geol. Survey Folio No. 64 (1900); "Chemical Analyses of Texas Rocks and Minerals," by E. P. Schoch, Bull. No. 1814, Univ. of Texas (1918); "Asphalt in the Anacacho Formation of Texas," by C. L. Baker, *Am. Assoc. Petrol. Geol. Bull.*, **12**, 995 (1928); "Quarrying Rock Asphalt in Texas," by C. H. Vivian, *Compressed Air Magazine*, **35**, 3244 (1930); "Rock Asphalt Mining in Texas," by J. B. Huttl, *Eng. Mining J.*, **133**, 225 (1932); "A Study of the Fluxing of the Bitumen Contained in Texas Limestone Rock-Asphalt," by J. T. L. McNew, Bull. No. 31, Texas Agr. and Mech. College Eng. Experimental Station (1933).

13 [p. 170] **U. S. Pats.** 452763 and 452764 of May 19, 1881 to Frederick Salathé; **Brit. Pat.** of 1891 (May 19), 8573 to J. Y. Johnson.

14 [p. 172] "A Contribution to the Chemistry of Some of the Asphalt Rocks Found in Texas," by H. W. Harper, Univ. of Texas Mineral Survey, Bull. No. 3 (May, 1902).

15 [p. 172] "Rock Asphalt Mining," by Gail Martin, *Eng. Min. J.*, **130**. 339 (1930).

16 [p. 173] "Note on a Specimen of Gilsonite from Uintah County, Utah," by R. W. Raymond, *Am. Inst. Mining Eng.*, **17**, 113 (1888); "Bituminous Sandstone Near Vernal, Utah," by E. M. Spieker, Bull. No. 822-C, U. S. Dept. Interior, Geol. Survey, Wash., D. C. (1930).

17 [p. 174] "The Asphaltum Deposits in California," by E. W. Hilgard, Mineral Resources of the U. S. 1883–4, U. S. Geol. Survey, Wash., D. C. (1885).

18 [p. 176] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 201, John Wiley & Sons, N. Y. (1908).

19 [p. 177] "Bituminous Sands of Northern Alberta," by S. C. Ells, Bull. No. 281, Dept. of Mines, Ottawa, Canada (1914); "Second Annual Report on the Mineral Resources of Alberta 1920," by J. A. Allan, Edmonton, Alberta (1921); "The Chemical Investigation of the Asphalt in the Tar Sands of Northern Alberta," by V. K. Krieble and W. F. Seyer, *J. Am. Chem. Soc.*, **43**, 1337 (1921); "Bituminous Sands of Alberta," by S. C. Ells, Canada Dept. Mines, Mines Branch, Summary Rept. 1920, Ottawa, Canada (1922); "Extent and Characteristics of Northern Alberta Bituminous Sands," by S. C. Ells, *Nat. Petroleum News*, **18**, 69 (1924); "Bituminous Sands of Northern Alberta," by S. C. Ells, Bull. No. 625, Dept. of Mines, Ottawa, Canada (1924).

20 [p. 180] Commerce Report No. 281, p. 948, Wash., D. C. (Nov. 30, 1920).

21 [p. 180] "The Geology of Cuban Petroleum and Asphalt Deposits," by E. de Golyer, *Bull. Am. Assoc. Petroleum Geologists*, **2**, 133 (1918); "Análisis de Minerales," Boletin de Minas, pp. 84–86, Havana, Cuba (Jan., 1918).

22 [p. 180] "On the Bituminous Deposits Situated at the South and East of Cardenas, Cuba," by S. F. Peckham, *Am. J. Science*, **12**, 33 (1901).

23 [p. 181] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 195, John Wiley & Sons, N. Y. (1908); "The Bitumens of Cuba," by Clifford Richardson.

24 [p. 182] "Report of the Inspector of Asphalt and Cement," Engineering Department, Dist. of Columbia, Wash., D. C. (1892); "On the Nature and Origin of Asphalt," The Barber Asphalt Paving Co., Long Island City, N. Y. (Oct., 1898); *J. Soc. Chem. Ind.*, **17**, 13 (1898); "The Proximate Composition and Physical Structure of Trinidad Asphalt," by Clifford Richardson, *Proc. Am. Soc. Testing Materials*, **6**, 509 (1906); "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 176 et seq., John Wiley & Sons, N. Y. (1908); "Trinidad and Bermudez Lake Asphalt and Their Use in Highway Construction," by Clifford Richardson (pamphlet), Phila., Pa. (1910); "The Wonderland of Trinidad," The Barber Asphalt Paving Co., Phila., Pa. (1910); "Studies in Asphalt," by C. J. Frankforter, *J. Ind. Eng. Chem.*, **2**, 239 (1910); "An Examination of Some Bituminous Minerals," by F. C. Garrett, *J. Soc. Chem. Ind.*, **31**, 314 (1912); "The Trinidad Asphalt Lake," by Edmund Graefe, *Z. angew. Chem.*, **26**, 233 (1913); *Chem. Ind.*, **35**. 175 (1912); "The Hydrocarbons of Utah," by Carlos Bardwell, et al., *J. Ind. Eng. Chem.*, **5**, 573 (1913); **6**, 805 (1914); "Production of Trinidad and Bermudez Lake Asphalts," by C. N. Forrest, *Roads and Streets*, **5**, 25–30 and 46–47 (1915); "Mining Trinidad Asphalt," by Clifford Richardson, *Eng. Mining J.*, **99**, 483 (1915); "The Lake Asphalt Industry," by J. S. Miller, Jr., *Chem. Met. Eng.*, **22**, 749 (1920); "Trinidad Lake Asphalt Mining," by H. T. McFarland, Salt Lake Mining Rev., **22**, 41 (1920); "The Chemistry of Bituminous Highway Construction—Native Asphalts," by J. S. Miller, Jr., *J. Soc. Chem. Ind.*, **48**, 782 (1929).

25 [p. 186] "A Unique Geophysical Phenomenon, Trinidad Asphalt, Interesting from the View of Dispersoid Chemistry," by Clifford Richardson, *J. Phys. Chem.*, **19**, 241 (1915); "Colloids and Their Occurrence in Trinidad Lake Asphalt," by J. S. Miller, Jr., *J. Eng. Club*, Baltimore, **6**, 131 (1916); "The Nature and Origin of Petroleum and Asphalt," by Clifford Richardson, *Eng. Chem.*, **8**, 4 (1916); *Met. Chem. Eng.*, **16**, 3 (1917).

26 [p. 187] "Asphalt, Bitumen and Tar: Their Respective Uses in Road Construction," by D. M. Wilson, *Chemistry and Industry*, **53**, 924 (1934).

27 [p. 187] "On the Nature of Parianite," by S. F. Peckham, *J. Franklin Inst.*, **149**, 161 (1900).

28 [p. 188] **U. S. Pat.** 1087085 of Feb. 10, 1915 to C. N. Forrest.

29 [p. 188] **U. S. Pat.** 1057667 of Apr. 1, 1913 to J. A. W. Pine and W. B. Ruggles.

30 [p. 189] "The Properties of Bitumen Poor and Rich in Paraffin," by J. Manheimer, *Petroleum Z.*, **28**, No. 16, 6 (1932).

31 [p. 190] "Petroliferous Rocks in Sierra da Baliza," by E. P. de Oliveira, *Amer. Inst. Min. Eng. Trans.*, **65**, 241 (1921).

32 [p. 190] "Petroleum Geology of the State of São Paulo," by C. W. Washburne, Commissão geographica e geologica do estado de São Paulo (Brazil), p. 147 (1930).

33 [p. 190] "Informe sobre los terrrenos petroliferos del departamento de San Rafael," by R. Zuber, Buenos Aires (1892).

34 [p. 191] "Ueber Erdöllagerstätten im Magdalena-Tal (Columbien)," by Otto Stützer, *Petroleum Z.*, **19**, 407 (1923).

35 [p. 191] "Erdöl- und Asphaltaustritte in Eruptivgesteinen und Kristallinen Schiefern des Mittleren Magdalenentals," by Otto Stützer, *Zeit. d. D. Geol. Ges.*, **75** (1923).

36 [p. 191] "Report on Asphalts in South-Central Part of Department of Boyacá," by E. Grosse, Bol. Minas y Petroleos (Bogota), **1**, 99–113 (1929).

37 [p. 191] "Oil Development in Ecuador During 1923," by J. H. Sinclair, *Am. Inst. Mining Eng.*, pp. 199–207 (1924).

38 [p. 191] "Die geologische Horizonte der fossilen Kohlen, usw.," by C. Zinken, p. 155, Leipzig (1883); "Etudes géologique sur l'asphalte et le bitume," *Bull. Soc. de Science Naturelles de Neuchâtel*, Departments of Haute-Savoie and Ain, Vol. 18 (1890); "Le Pétrole, l'Asphalte et le Bitume," by A. Jaccard, Paris, Librairie Germer Baillière et Cie, p. 112 (1895); "Asphalt Deposits in France," by P. Nicou, *Annales des Mines*, **10**, 513 (1906); "Asphalt Industry of France," by V. Charrin, *Génie civil*, **108**, 323 (1936).

39 [p. 192] "Bitumen," by H. W. Halleck, p. 30 et seq, Peter Force, Wash., D. C. (1841); S. P. Pratt, *Quarterly J. Geol. Society* (London), **2**, 8 (1846).

40 [p. 192] "Asphalt-Limestone Deposits in the Concessions of Servas and St. Jean-de-Marvéjols," by M. Coignard, *Ann. des Mines*, **11**, Book 5, 594 (1907).

41 [p. 192] "Composition of Bituminous Limestone from Lovagny," by H. Gault and L. Boisselet, *Mat. Grasses*, **14**, 6156 (1922).

42 [p. 192] C. Puvis, *Ann. d. Mines*, **6**, No. 3, 179 (1834); A. Rozet, *Bull. Soc. Géol.* France, **7**, 136 (1835); A. Ure, *London J. Arts*, **14**, No. 3, 251 (1839).

43 [p. 194] "Asphalts and Allied Products." by H. Rabaté, *Chim. et Ind.*, **29**, 766 and 1026 (1933).

44 [p. 194] "A Treatise on Petroleum," by Boverton Redwood, 4th Edition, Vol. I, p. 179 (1922); "The Asphaltic Limestone Mine of Pont-du-Château," by E. Aumaitre and M. Roques, Congr. intern. mines mét. géol. appl., 7e Session, Paris (Oct. 1935); Géol. **1**, 511 (1935).

45 [p. 195] "Etudes Géologiques sur l'Asphalte et la Bitume," by A. Jaccard, *Bull. Soc. des Sciences Natur de Neuchâtel*, Vol. XVII (1890); "Le Pétrole, l'Asphalte et la Bitume," by A. Jaccard, Librairie Germer Ballière et Cie, Paris, p. 122 (1895); "The Asphalt Mine in the Val de Travers," by Ernst Rüst, *Schweiz Chem. Ztg.*, **1**, 49 (1917); *Annales des Mines*, 11e *Serie*, **2** (1912).

46 [p. 196] "Historique de Péchelbronn," by Paul de Chambrier, Attinger Frères, Paris (1919); "The Mines and the Refinery of Péchelbronn," by Paul and Jacques de Chambrier, *Mat. grasses*, **13**, 5784 (1921).

47 [p. 197] "Petroleumindustrie Oesterreich-Deutschlands," by Leo Strippelmann Verlag von G. Knapp, Leipzig (1878–9); "Vorkommen und Ursprung des Petroleums," by Nöldecke, A. Schulze, Celle und Leipzig (1883); "Oelheim und Beiträge zur Geologie und Paläontologie des Herzogtums Braunschweig, usw.," by Alwin Freystedt, Vol. I, Braunschweig (1894).

48 [p, 197] "Mineral-asphaltstein der Mine zur Limmer, Feldmark Velber bei Hannover; nebst eine kurzen Anleitung zur Anwendung desselben bei verschiedenen Bauten," by D. H. Henning, Verfasser, Limmer (1866); "Der Asphalt," by Ernst Nöthling (W. Jeep), p. 7, Leipzig (1899).

49 [p. 198] "Die Asphalt-Industrie bei Escherhausen im Kreise Holzminden," by Ulrich Söhle, *Der Steinbruch*, **9**, 410, 424, 442 and 453 (1914); "Deutscher Asphaltbergbau bei Escherhausen," by Neubauer, *Zeitsch. für das Berg-Hütten-und Salinenwesen*, **79**, 167 (1931); "Deutscher Asphaltbergbau," by Anon., *Teer u. Bitumen*, **30**, 385 (1932).

50 [p. 201] A. Steur, *Notizbl. Ver. f. Erdkunde u. Groszh. geol. Landesanst.*, **25**, No. 4, 35 (1905); "Occurrence of Asphalt at Mettenheim," by A. Steuer, *Chem. Zentr.*, **36**, 986 (1907).

51 [p. 201] "Asphaltgänge im Quarzporphyr von Dossenheim bei Heidelberg," by W. Salomon, *Berichte über die Versammlungen des oberrheinischen geologischen Vereins*, 42nd Versammlung, p. 116, Karlsruhe (1909).

52 [p. 201] "Das Erdöl," by Engler-Höfer, 2nd Edition, Vol. 2, Part 2, p. 276, Verlag S. Hirzel, Leipzig (1930).

53 [p. 202] "Die Enstehung des Erdöles, verwandter Kohlenwasserstoffe und gewisser Kohle," by P. Krusch, XV Int. Geol. Congr. South Africa (1929); *Comptes Rendus*, **2**, 294 (1930).

54 [p. 202] "Jelić Analisi dei cinque Principali Asfalti Dalmati," by Giorgio Drazoević, *Verh. k. k. geol. Reichs-Anst.*, p. 109 (1885); Jahrb. k. k. geol. Reichs-Anst. (Vienna), **7**, 761 (1856); Oesterr. zeitschr. f. Berg-u. Hüttenwes., **47**, 523 (1899); Montanzeitung f. Oesterr.-Ungarn **7**, 225, (1900); *Verh. k. k. geol. Reichs-Anst.*, **10** (1902); "Ursprung, Vorkommen und Beschaffenheit der dalmatinischen Asphaltlagerstätten," by F. K. von Marilaun, *Petrol. Zeitschr.*, **15**, 489, 549 and 629 (1920).

55 [p. 203] "Asphaltkalke in Hercegowina," by Friedrich Katzer, *Globus*, **83**, No. 12 (1903); "Geologischer Führer durch Bosnien und die Hercegowina," p. 256, Sarajevo (1903); "Ueber den heutigen Stand der geolog. Kenntnis Bosniens-Hercegowinas," *Compt. rend. Congrès géol. Internat.*, **9**, 337 (1904).

56 [p. 203] G. A. Koch, *Mitteilg. naturwiss. Ver. Steiermark*, **26**, 141 (1889).

57 [p. 203] "Die Tiroler Asphaltschiefer-Vorkommen," by M. Isser, *Petroleum Z.*, **10**, 578 (1915); *Montan Rundsch.*, **7**, 267 (1915); *Chem. Ztg.*, **37**, 447 (1915).

58 [p. 204] "Chemische Analyse des Asphaltes der Muraköz und des von Hagymádfalu," by Nendtvich Károly, *A. m. kir. Természettudományi Társulat Evkönyvei*, **1**, 16 (1841–1845); "Die Tataroser Petroleum- und Asphaltgesellschaft," by Anon., *Ungar. Montanind. Ztg.*, p. 157 (1889); "Ungarischer Asphaltaktiengesellschaft in Mezötelegd und Felsöderna," by Anon., *Ungar. Montanind. Ztg.*, **21** (1894); *Allg. österr. Chem. u. Techn. Ztg.*, **21** (1894); "Der Asphaltbergbau in Tataros und die Aufbereitungswerke," by Kauffmann Kamillo, Bányászati és Kohászati Lapok, p. 228 (1897); "Ueber die Asphaltlager im Komitate Bihar," by Szontagh Tamas, Bányászati és Kohászati Lapok, p. 248 (1897); "Petroleum und Asphalt in Ungarn," by Theodor Posewitz, Buchdruckerei des Franklin-Vereins, Budapest, pp. 411–415 (1907).

59 [p. 204] "Chemische Zusammensetzung und Unterscheidung der natürlichen und künstlichen Asphalte," by Julius Marcusson, *Chem. Rev. Fett-u. Harz-Ind.*, **19**, 166 (1912).

60 [p. 204] "Occurrence of Asphalt at Strečno (Slovakia)," by J. J. Jahn, *Petroleum Z.*, **19**, 219 (1923).

61 [p. 204] "Natural Asphalt from Strečno in Slovakia," by M. Landova, *Chem. Listy*, **24**, 461 (1930).

62 [p. 206] "Die Chemie und Technologie der Natürlichen und Künstlichen Asphalte," by Hippolyt Köhler and Edmund Graefe, p. 58, Friedr. Vieweg & Sohn, Braunschweig (1913); "Chemical-technical Investigation of Matitza Asphalt," by C. Nicolescu-Ottin, *Chem. Zentr.*, **2**, 504 (1915); "Products Obtained by the Dry Distillation of Matitza Asphalt," by C. Nicolescu-Ottin, *Bull. Acad. Roumaine*, **5**, 129 (1916–1917); "Matitza Asphalt," by C. Nicolescu-Ottin and S. Savenco, *Petroleum*, **29**, 1 and 13 (1933).

63 [p. 206] "Variae Historia," by Claudius Aelianus, Vol. XIII, §16; "Voyage dans la Grèce," by Ponqueville, Vol. I, p. 271.

64 [p. 206] "Notes sur les Sources et Mines d'Asphalt en Bitume Mineral de la Grèce," by M. Virlet, *Bull. Soc. Geol. France*, **2**, 203 (1834); "Description Géologique des Gisements Bituminifères et Pétrolifères de Sélenitza dans l'Albanie," by H. Coquand, *Bull. Soc. Geol. France*, **25**, Series II, 20 (1868); "The Properties of Bitumen Poor and Rich in Paraffin," by J. Manheimer, *Petroleum Z.*, **28**, No. 16, 6 (1932).

65 [p. 206] "Petrolio di Toccoe Bitumi di Letto Monopolo," by G. Capellini, Torino (1866); "Gisements de Bitumes, Pétroles et de Divers Minéraux Provinces Grete et de Fuosinone," by L. Bidou, Sienne (1878); "L'Industrie des Hydrocarbures en Italie," by E. Camerana, Rome (1907); "The Asphaltic Limestone Region of the Pescara Valley," by Georg Thiel, *Z. prak. Geol.*, **20**, 169 (1912).

66 [p. 208] "L' Industria dell' Asfalto," by Ernesto Ascione, Francesco Vallardi, Milan (1913).

67 [p. 209] "Asphaltic Rock in Italy," *Oil and Gas J.*, **22**, 117 (Aug. 16, 1923); "The Asphaltic Mines of Ragusa," by Gaetano Castelli, *Rassegna Min. Met. Chim.*, **58**, 136 (1923); "Studi in Sicilia," by E. Cortesi, *Carta Geol. d'Italia, Memorie*, Vol. 20 (1926); "Sulla geologia degli idrocarburi della Sicilia ancha in raporto colla formazione degli zolfi," by Ramiro Fabiani, Boll. Assoc. Min. Sic., No. 3, Palermo (1927); "Italian Asphaltic Rocks Used in Road Construction," by A. Di Renzo, *Le Strade*, **16**, 175 (1934); "Idee sulla provenienza degli idrocarburi di Sicilia," by Ramiro Fabiani, XV Int. Cong., South Africa (1929); *Comptes Rendus*, **2**, 303 (1930).

68 [p. 210] "Dry Distillation of Natural Sicilian Asphalt and Bitumen," by Italian Gas Society (Turin), *Ind. Chim.*, **13**, 103 (1913); "The Asphalt Mines at Ragusa," by Gaetano Castelli, *Rass. Min. Met. Chim.*, **58**, 136 (1923); "Sources of Mineral Oils Available to Italy," by Camillo Manuelli, *Atti. Congresso naz. Chim. Ind.*, 182 (1923); 201 (1924); *Indust. olii e grassi*, **3**, 18 (1923); "Asphalt bei Ragusa auf Sizilien," by Dr. Ing. Nehring, *Teer u. Bitumen*, **29**, 315 (1931).

69 [p. 212] "Ueber den griechischen Asphalt und seine technische Bedeutung," by A. C. Vournasós, *Dingler's poly. J.*, **321**, 200 (1906); "Sur la Zone Hydrocarburée de la Grèce Occidentale," by C. A. Kténas, *Comptes Rendu.*, **170**, 737 (1920).

70 [p. 212] "Historiarum," by Herodotus, Vol. I, 119 and 179; Vol. IV, 195; "De Architectura," by Vitruvius, VIII, 3 and 8; "Historiae Naturalis," by Pliny (the Elder), XXXV, 15 and 178; "Materia Medica," by Dioscorides, I, 99; "Zante," by Louis Salvator, p. 179, L. Wœrl. Leipzig (1904).

71 [p. 213] "Die geologische Horizonte der fossilen Kohlen. Die Vorkommen der fossile Kohlenwasserstoffe," by C. Zinken, Leipzig, p. 153 (1883); "Ueber die Petroleumlager von Portugal," by Eugen Ackermann, *Chem.-Ztg.*, **32**, 460 (1908).

72 [p. 213] "Die Mineralfundstätten der iberischen Halbinsel," by Tenne-Calderón, Berlin (1902).

73 [p. 214] "Exploitación de las Minas de Asfalto de Fuentetoba-Cidones," by R. Otlet, *Revista Minera*, **66**, 51 (1915).

74 [p. 214] "Die Asphaltvorkommen Russlands," by A. F. v. Stahl, *Asphalt u. Teerindustrie-Zeitung*, **26**, 617 and 644 (1916).

75 [p. 215] "Syzran Asphalts," by A. Parschin, *Chem. Zentr.*, **1**, 2270 (1926).

76 [p. 215] "New Deposits of Asphaltum," by K. Charitschkow, *Chem. Zeit.*, **31**, Rep. 116 (1907).

77 [p. 216] "Russian Gilsonite," by K. P. Likhushin and T. Y. Nosovskaya, *Azerbaĭdzhanskoe Neftyanoe Khozyaĭstvo*, **3**, 84 (1934).

78 [p. 216] "Sur la composition de l'asphalte ou bitume de Judée," by B. Delachanel, *Comp. rend. Acad. science*, **97**, 491 (1883); "Die Mineral-Industrie in der Turkei," by Anon., *Z. für angew. Chem.*, **16**, 901 (1903); "Oil and Asphalt in Palestine, Syria and Mesopotamia," by Felix Oswald, *Petroleum World*, **15**, 459 (1918); "Die Asphaltlager von Lattakia," by Eduard Marckwald, *Petroleum Z.*, **18**, 165 (1922).

79 [p. 218] "The Mineral Resources of Syria," by I. M. Toll, *Eng. Mining J.*, **112**, 846 (1921); "Syrian Asphalt," by Friedrich König-Hietzing, *Petroleum Z.*, **17**, 1259 (1921); "Die Asphaltlager von Latakia," by Eduard Marckwald, *Petroleum Z.*, **18**, 165 (1922).

80 [p. 218] "Turkish Bitumens," by Anon., *J. Roy. Soc. Arts*, **56**, 829 (1908).

81 [p. 218] "Asphalt Mines in Syria," Reports from the Consuls of the U. S., **42**, 153 and 228 (1893); "Untersuchung über Syriche Asphalte," by Friedrich König-Hietzing, *Petroleum Z.*, **17**, 1259 (1921).

82 [p. 219] "Turkish Bitumens," by Anon., *J. Roy. Soc. Arts*, **56**, 829 (1908); "Die Mineralschätze der Turkei," by C. Doelter, *Chem. Zeit.*, **11**, 108 (1916); Montan Rundschau, **8**, 217 (1916); "The Mineral Resources of Syria," by I. M. Toll, *Eng. Min. J.*, **112**, 846 (1921); "Das Erdölvorkommen in Türkisch-Armenien," by A. F. v. Stahl, *Petroleum Z.*, **23**, 115 (1927); "Untersuchung der Bitumenvorkommen in Nahren Osten," by Rudolf Seiden, *Chem. Z.*, **53**, No. 35, 341 and No. 36, 362 (1929).

83 [p. 220] "Fuel in Turkey," by Leon Dominian, *Bull. Am. Inst. Mining Eng.*, **109**, 1022–30 (1916); "Geology of Mesopotamia and its Borderlands," by Naval Intelligence

Division, Naval Staff of Admiralty of Great Britain, Chap. VIII, Part 2, pp. 72 and 91 (1921).

84 [p. 220] "Mineralvorkommen Anatoliens," by F. Frech, *Glückauf*, **51**, 464–470 (1915); "Die Mineralschätze der Turkei," by C. Doelter, *Montan Rundschau*, **8**, 217 (1916); *Chem. Zentr.*, **2**, 108 (1916); "Das Erdölvorkommen an der türkisch-persischen Grenze," by F. Frech, *Geogr. Zeitsch.*, **21**, 9 (1921).

85 [p. 220] "Asphalt Deposits in Bahrein Island," by Anon., *Bull. Imperial Inst.*, **1**, 116 (1906); "Asphaltic Rock on the Island of Bahrein in the Persian Gulf," by Hans Höfer, *Chem. Zentr.*, **36**, 987 (1907).

86 [p. 221] "Aus der ältesten Geschichte des Bitumens," by R. J. Forbes, *Bitumen*, **4**, Nos. 1, 2 and 3 (1934).

87 [p. 221] "Occurrence of Bitumen in Bombay Island," by C. S. Fox, *Rec. Geol. Survey of India*, **54**, 117 (1922).

88 [p. 221] Private communication from Prof. T. Takakuwa, of the Kiriu Technical College, Kiriu, Japan; "Occurrence of Asphalt in the Ugo Province," by Ryohei Katayama, *Beitr. Mineral* (Japan), **5**, 303 (1915).

89 [p. 223] "Geological Memoranda," by Walter Howchin, *Trans. and Proc. Royal Soc.*, New South Wales, **43**, 45 (1911).

90 [p. 223] "The Reputed Petroliferous Area of the Warren River District (Southwest Division)," by H. P. Woodward, Bull. No. 65, Western Australia Geol. Survey, F. W. Simpson, Govt. Printer, Perth (1915); "Petroleum Prospects, Kimberly District of Western Australia and Northern Territory," by Commonwealth of Australia, pp. 31 and 37 (1924); "The Search for Oil in Australia," by Arthur Wade, *Inst. Petroleum Tech. J.*, **12**, 155 (1926).

91 [pp. 223, 224] "Reconnaissance of Country between Recherche Bay and New River, Southern Tasmania," by W. H. Twelvetrees, Bull. No. 24, Tasmania Geol. Survey, J. Vail, Govt. Printer, Hobart (1915).

92 [p. 224] "Boeton Asphalt," by Anon., *Quarry & Roadmaking*, **36**, 221 (1931); "Asphaltbitumen en Teer," by F. J. Nellensteyn and R. Loman, p. 140, Amsterdam (1932); "Het Onstaan van Asfalt-Bitumen," by Dirk Thöenes, Drukkerij Waltman, Delft (1936).

93 [p. 226] "Die Bitumenlager von Sidi Messaoud in Algier," by Edmund Graefe, *Petroleum Z.*, **10**, 641 (1915).

94 [p. 226] "Mineral Oil, Solid Bitumen, Natural Gas and Oil Shales," by P. A. Wagner, *South African J. Ind.*, **1**, 126 (1917); **3**, 29 (1917); "The Pearl Bitumen Co.'s Prospects," by Anon., *South African Mining & Eng. J.*, **29**, Part. I, 439 (1920).

95 [p. 226] "The Bituminous Sandstones of Madagascar," by J. A. Hardel et al., *Chimie et Industrie*, **11**, 1003 (1924).

CHAPTER X

1 [p. 227] "The Natural Hydrocarbons, Gilsonite, Elaterite, Wurtzilite, Grahamite, Ozokerite and Others," by R. B. Ladoo, Bureau of Mines, Wash., D. C. (May, 1920); "Hydrocarbon Wealth of the State of Utah," by J. B. Jenson, *Salt Lake Min. Rev.*, **25**, 11 (1923).

2 [p. 227] "Gilsonite or Uintaite," by J. M. Locke, *Trans. Am. Inst. Mining Eng.*, **16**, 162 (1887); "Notes on a Specimen of Gilsonite from Uinta County, Utah," by R. W. Raymond, *Trans. Am. Inst. Mining Eng.*, **17**, 113 (1888); "Nature of Uintaite," by Dr. Henry Wurtz, *Eng. and Mining J.*, **48**, 114 (1889); "The Uintaite (Gilsonite) Deposits of Utah," by G. H. Eldridge, 17th Annual Report U. S. Geol. Survey, Wash., D. C., p. 915 (1896); "Investigation of Utah Gilsonite—A Variety of Asphalt," by W. T. Day, *J. Franklin Inst.*, **140**, 221 (1895); **160**, 221 (1896); 22nd Annual Report U. S. Geol. Survey, by G. H. Eldridge, Part. I, pp. 327 and 340, Wash., D. C. (1901); "The Production of Asphalt, Related Bitumens and Bituminous Rock in 1910," by D. T. Day, p. 6, U. S. Geol. Survey, Wash., D. C. (1911); "Modern Asphalt Pavement," by Clifford Richardson, p. 208, John Wiley & Sons, New York (1908); "The Hydrocarbons of Utah," by Carlos Bardwell, et al., *J. Ind. Eng. Chem.*, **5**, 973 (1913); **6**, 865 (1914); "Gilsonite and Grahamite," by Clifford Richardson, *J. Ind. Eng. Chem.*, **8**, 496 (1916); "The Mining of Gilsonite in Utah," by W. J. Fene, Circ. No. 6069, Bureau of Mines, Wash., D. C. (May, 1928); "Gilsonite and Related Bitumens,"

by H. M. Langton, *Ind. Chemist*, **5**, 324 and 383 (1929); "Gilsonite Deposits of the Uintah Basin," by John Bristol, *Mining J.* (Arizona), **13**, 5 and 63 (1929).

3 [p. 230] **U. S. Pats.** 361347 of Apr. 19, 1887 to C. T. Crowell; 361759 of Apr. 26, 1887 to S. H. Gilson; 362076 of May 3, 1887 to S. H. Gilson; 415864 of Nov. 26, 1889 to S. H. Gilson.

4 [p. 230] **U. S. Pats.** 1573764 and 1573765 of Feb. 16, 1926 to C. N. Forrest.

5 [p. 230] **U. S. Pats.** 1459328 of Jun. 19, 1923 to C. N. Forrest, H. P. Hayden and O. R. Douthett; 1578235 of Mar. 23, 1926 to C. N. Forrest, H. P. Hayden and O. R. Douthett.

6 [p. 230] **U. S. Pat.** 1524859 of Feb. 3, 1925 to C. N. Forrest, H. P. Hayden and O. R. Douthett.

7 [p. 235] "Native Asphalts in Oregon," by E. T. Hodge, *Bull. Am. Assoc. Petroleum Geologists*, **11**, 395 (1927).

8 [p. 236] "The Russian Asphalt Gilsonite," by M. M. Faingar, O.M.T.I. Gorno-Geol.-Neftyanoe Izdat., "Crude Oils, Bitumens and Gases from Non-Caucasian Deposits," p. 61 (1934).

9 [p. 237] "Natural Pitch or Manjak from Trinidad," by T. A. Henry, Bull. Imp. Institute (Gt. Britain), **1**, 180 (1903); "Manjak as Worked at the Vistabella Mine, Trinidad," by J. C. T. Raspass, *Inst. Mining Eng.*, **36**, 119 (1909); "Manjak," by R. H. Emtage, *Mining Mag.*, **25**, 156 (1921).

10 [p. 238] "Manjak," by W. Merrivale, *Fed. Inst. Eng.*, **14**, 539 (1896); **16**, 33 (1898); "Results of the Analysis of Samples of New Zealand Coal and Ambrite, and of Barbados Manjak," by P. P. Bedson, *Institution Mining Eng.*, **16**, 388 (1899); "Die Bitumen von Barbados," by G. Du Bois, *Z. Prakt. Geol.*, **7**, 397 (1899); "Ueber das Vorkommen bituminöser Producte auf der Insel Barbados," by Alfred Rehwagen, *Berg.- u. Hütt.-Ztg.*, **60**, 467 (1901); "The Mining of Non-Metallic Minerals," by R. H. Emtage, *J. Royal Soc. Arts*, **52**, 367 (1904); "An Examination of Some Bituminous Minerals," by F. C. Garrett, *J. Soc. Chem. Ind.*, **31**, 314 (1912); "Properties and Uses of Manjak," by W. C. Phalen, Reports of Investigations, Bureau of Mines, Wash., D. C. (Nov. 1919); Commerce Report No. 252, p. 541 (Oct. 1919).

11 [p. 241] "Uranium-Bearing Asphaltite Sediments of Utah," by F. L. Hess, *Eng. Mining J.*, **114**, 272 (1922).

12 [p. 241] "Notes on Minor Occurrences of Oil, Gas and Bitumen with Igneous and Metamorphic Rocks," by Sidney Powers, *Bull. Amer. Assoc. Petroleum Geologists*, **16**, 837 (1932).

13 [p. 241] "Asphalt Mines in Colombia, South America," by Anon., *J. Soc. Chem. Ind.*, **23**, 278 (1904).

14 [p. 242] "The Asphalt of Bentheim," by Carl Engler and Leo Streppelmann, *Chem. Zentralblatt*, **15**, 271 (1884); "Bentheim Asphalt Deposits," by F. Rainer, *Petroleum Z.*, **22**, 677 (1926).

15 [p. 242] "The Mineral Resources of Syria," by I. M. Toll, *Eng. Mining J.*, **112**, 846 (1921); "Die Asphaltlager von Lattakia," by Eduard Markwald, *Petroleum Z.*, **18**, 165 (1922).

16 [p. 243] "Turkish Bitumen," by Anon., *J. Royal Soc. Arts*, **56**, 829 (1908).

17 [p. 244] "Grahamite, A Solid Native Bitumen," by Clifford Richardson, *J. Am. Chem. Soc.*, **32**, 1032 (1910); "Gilsonite and Grahamite, the Result of the Metamorphism of Petroleum under a Particular Environment," by Clifford Richardson, *J. Ind. Eng. Chem.*, **8**, 493 (1916).

18 [p. 245] **U. S. Pat.** 1332359 of Mar. 2, 1920 to R. R. Rosenbaum; 2069314 of Feb. 2, 1937 to J. K. Hunt and J. H. Shipp.

19 [p. 245] **U. S. Pat.** 100269 of Mar. 1, 1870 to E. J. de Smedt.

20 [p. 245] **U. S. Pat.** 101594 of Apr. 5, 1870 to E. J. de Smedt.

21 [p. 245] "Coal or Asphalt Vein of Wood County, West Virginia," by J. P. Leslie, *Am. Phil. Soc.*, **9**, 183 (1863); "Grahamite," by Henry Wurtz, *Am. J. Sci.*, No. 2, **42**, 420 (1865); *Am. Soc. Adv. Sci.*, **18**, 124 (1869); "On the Probable Origin of Albertite and Allied Minerals," by S. F. Peckham, *Am. Gas Light J.*, **11**, 164 (1869); "Uintaite, Albertite, Grahamite and Asphaltum, Described and Compared, with Observations on Bitumen and its Compounds," by W. P. Blake, *Am. Inst. Mining Eng.*, **18**, 563 (1889); "Notes on the West

Virginia Asphaltum Deposit," by W. M. Fontaine, *Am. J. Sci.*, 6, 409 (Oct. 14, 1873); "Note on the Occurrence of Grahamite in Texas," by E. T. Dumble, *Am. Inst. Mining Eng.*, 21, 601 (1892–1893); "Origin of Grahamite," by I. C. White, *Bull. Geol. Soc. Am.*, 10, 277 (1898).

22 [p. 247] "Grahamite, A Solid Native Bitumen," by Clifford Richardson, *J. Am. Chem. Soc.*, 32, 1032 (1910); "Chemical Analyses of Texas Rocks and Minerals," by E. P. Schoch, *Bull. Univ. Texas, No. 1814* (1918).

23 [p. 247] "Grahamite Deposits of Southeastern Oklahoma," by J. A. Taff, Contributions to Economic Geology, Bull. No. 380, U. S. Geol. Survey, p. 286, Wash., D. C. (1908); "Asphalt and Petroleum in Oklahoma," by L. L. Hutchison, Bull. No. 2, Okla. Geol. Survey, Guthrie, Okla. (1911).

24 [p. 250] "Variations of the Physical Characteristics of a Petroleum Residue with Increasing Percentages of Grahamite," by H. Rossbacher, *J. Ind. Eng. Chem.*, 7, 205 (1915).

25 [p. 252] "Asphaltic Coals in the Indian Territory," by W. R. Crane, *Mines and Minerals*, 26, 252 (1906).

26 [p. 254] "On a Deposit of Grahamite, Known as the Christo Coal Mine; 100 Miles Southwest of the Port of Tampico, Mexico," by J. P. Kimball, Pamphlet published by the Author, 49 Wall St., New York (May 15, 1876); "Occurrence of Grahamite at the Cristo Mine in the Huasteca, Vera Cruz, Mexico," by J. P. Kimball, *Am. J. Sci.*, 12, 277 (1876).

27 [p. 254] "Bitumen in Cuba," by T. W. Vaughan, *Eng. Mining J.*, 73, 344 (1902); "Mineral Resources of Cuba in 1901," Gussenheimer, Weil & Co. (1902); "The Bitumens of Cuba," by Clifford Richardson (1910); "An Examination of Some Bituminous Minerals," by F. C. Garrett, *J. Soc. Chem. Ind.*, 31, 314 (1912).

28 [p. 255] "Mining Asphalt in Cuba," by A. E. Northey, *Eng. Min. J.*, 132, 66 (1931).

29 [p. 256] "Manjak," by R. J. L. Guppy, *Geol. Mag.*, 1, 276 (1904); "The San Fernando Manjak Field," Council Papers No. 3 (1905); Nos. 35 and 130 (1906), Govt. Geologist, Port-of-Spain, Trinidad; "Manjak as Worked at the Vistabella Mine, Trinidad," by J. C. T. Raspass, Trans. Inst. Mining Eng., Newcastle-upon-Tyne (Sep. 7, 1908); "An Examination of Some Bituminous Minerals," by F. C. Garrett, *J. Soc. Chem. Ind.*, 31, 314 (1912); "Manjak," by Anon., *Mining and Sci. Press*, 3, 36 (1915).

30 [p. 259] "Vanadium Minerals in Argentine," by G. Fester and F. Bartuzzi, *Z. angew. Chem.*, 38, 363 (1925).

31 [p. 261] "Vanadium Deposits in Peru," by D. F. Hewett, *Am. Inst. Mining Eng.*, 40 (1909); "The Vanadiferous Asphaltites of Central Peru," by J. C. Baragwanath, *Eng. Mining J.*, 111, 778 (1921); "Geologie von Peru," by G. Steinmann, Heidelberg (1929).

CHAPTER XI

1 [p. 262] "The Natural Hydrocarbons: Gilsonite, Elaterite, Wurtzilite, Grahamite, Ozokerite and Others," by R. B. Ladoo, Bureau of Mines, Wash., D. C. (May, 1920).

2 [p. 262] "The Asphalt and Bituminous Rock Deposits of the United States," by G. H. Eldridge, 22nd Annual Report, U. S. Geol. Survey, Part I, pp. 222 and 360, Wash., D. C. (1901).

3 [p. 263] M. Lister, *Phil. Trans.*, 6179 (1673).

4 [p. 263] C. Hatchett, *Linn. Trans.*, 4, 146 (1797).

5 [p. 263] H. J. Klaproth, *Beitr.*, 3, 107 (1802).

6 [p. 263] "Elaterite," by W. Morrison, *Mining Mag.*, 8, 133 (1889); "The Hydrocarbons of Eastern Utah, with Special Reference to the Deposits of Ozokerite, Gilsonite and Elaterite," by Don Maguire, *Mines and Minerals*, 20, 398 (1900).

7 [p. 263] C. T. Jackson, *Pharm. J.*, 31, 763 and 785 (1872); G. C. Morris, *Proc. Acad. Phila.*, 131 (1877); A. C. Cumming, *Proc. Royal Soc. Vict.*, 15, 134 (1903); L. A. Boodle, *Bull. Roy. Botanical Gardens, Kew*, 145 (1907); "Coorongite, a Petroleum Product," by E. de Hautpick, *Mining J.*, 142, 575 (1923).

8 [p. 263] "Ueber das Elaterit aus dem Gebiet Semirjetschensk," by M. A. Rakusin, *Petroleum Z.*, 8, 729 (1913).

9 [p. 264] "The Occurrence of Coorongite in Central Asia," by R. J. Morgan, *Chem. Eng. Mining Rev.*, **13**, 348 (1921).

10 [p. 264] "Wurtzilite from the Uintah Mountains, Utah," by W. P. Blake, *Eng. Mining J.*, **48**, 542 (1889); **49**, 59 (1890); *Am. Inst. Mining Eng.*, **18**, 497 (1889); "Recent Uses of Utah Elaterite," by Elihu Palmer, *Eng. Mining J.*, **102**, 856 (1916); "Chemical Properties of Utah Hydrocarbons," by Carlos Bardwell, B. A. Berrymen, T. B. Brighton and K. D. Kuhre, *Trans. Utah Acad. Sci.*, **1**, 78 (1918).

11 [p. 266] "Petroleum and Coal," by Julius Marcusson, *Chem. Ztg.*, **42**, 437 (1918).

12 [p. 267] "Reports on the Albert Coal Mine," by C. T. Jackson, *Proc. Boston Soc. Natural History*, **3**, 279 (1851); *Am. J. Sci.*, **13**, 2 (1851); "Albert Coal," by C. M. Wetherill, *Am. Phil. Soc. Phila.*, 353 (1852); "Albertite," by J. W. Dawson, Acadian Geol., p. 198, Edinburgh (1865); "Albertite," by C. H. Hitchcock, *Am. J. Sci.*, **39**, II, 267 (1865); "Uintaite, Albertite, Grahamite, and Asphaltum, Described and Compared, with Observations on Bitumen and its Compounds," by W. P. Blake, *Am. Inst. Mining Eng.*, **18**, 563 (1889); "Albertite," by A. Milner, *J. Mining Soc. Nova Scotia*, **17**, 62 (1912); "Oil Shales of America," by Charles Baskerville and W. A. Hamor, *J. Ind. Eng. Chem.*, **1**, 507 (1909); **5**, 73 (1913).

13 [p. 269] "The Bitumen of the Falkland Islands," by Anon., *Petroleum Rev.*, **27**, 304 (1913).

14 [p. 269] "Bentheim Asphalt," by Carl Engler and Leo Streppelmann, *Ding. poly. J.*, **250**, 216 and 265 (1883); "Bentheim Asphaltite as a Valuable Paint," by Hans Bockshammer and Fritz Kurzen, *Teer u. Bitumen*, **32**, 258 (1934).

15 [p. 270] "On Tasmanite, a New Mineral of Organic Origin," by A. H. Church, *Phil. Mag.*, **28**, 465 (1864); "Tasmanite," by E. T. Newton, *Geol. Mag.*, **2**, 336 (1875); "Tasmanite, or Mersey Shale," by E. T. Newton, *Proc. Royal Soc. Tasmania*, **5**, 4 (Mar. 1876).

16 [p. 270] P. Gomes, *Comm. Dir. Trabalhos Geol.*, Portugal, **3**, 244; **4**, 206 (1896–8); "Peculiar Phases of Oil Saturation in Certain Sandstones," by G. M. Ruby, *Bull. Am. Assoc. Petroleum Geologists*, **7**, 478 (1923).

17 [p. 271] "Grahamite Deposits Found in Eastern Oklahoma," by J. A. Taff, Bull. No. 380, U. S. Geol. Survey, Wash., D. C., p. 295 (1909).

18 [p. 271] "Arkosite," by S. P. Thacher, *J. Ind. Eng. Chem.*, **5**, 83 (1913).

19 [p. 272] "An Occurrence of Asphaltite in Northeastern Nevada," by Robert Anderson, Bull. No. 380, U. S. Geol. Survey, Wash., D. C., p. 283 (1909).

20 [p. 272] "Bitumen in Nonesuch Formation of Keweenawan Series of Northern Michigan," by C. G. Carlson, *Bull. Am. Assoc. Petroleum Geologists*, **16**, 737 (1932).

21 [p. 272] "Vanadium Deposits in Peru," by D. F. Hewett, *Am. Inst. Mining Eng.*, **40**, (1909); "The Vanadiferous Asphaltites of Central Peru," by J. G. Baragwanath, *Eng. Mining J.*, **111**, 778 (1921); "The Vanadiferous Bitumens of Peru," by Anon., *Petroleum Times*, **10**, 432 (1923); "Geologie von Perû," by G. Steinmann, Heidelberg (1929).

22 [p. 273] "Memoir upon the Composition of Bitumens," by J. B. Boussingault, *Annales de Chemie et de Physique*, **64**, 141 (1837).

23 [p. 273] "Report on the Coal Measures and Associated Rocks of South Brazil," by I. C. White, Report made by the Coal Commission to the Brazilian Government, Rio de Janeiro (1907).

24 [p. 274] "An Occurrence of Impsonite in West Australia," by R. F. Farquharson, *Chem. Eng. Mining Rev.*, **14**, 407 (1922).

CHAPTER XII

1 [p. 275] "Oil Shales of the Lothians," Part I: "Geology of the Oil Shale Fields," by H. M. Cadell and I. S. Grant Wilson; Part II: "Methods of Working Oil Shales," by W. Caldwell; Part III: "Chemistry of the Oil Shales," by D. R. Steuart; issued by Dept. of Mines, Geol. Survey, Scotland (1906); "Mineralogy of Nova Scotia," by H. How, Canadian Dept. of Mines, Bull. No. 1072, p. 132 (1908); Bull. No. 1120, p. 200 (1909); "Joint Report of the Bituminous Oil-shales of New Brunswick and Nova Scotia," Canadian Dept. of Mines,

Bulls. 55 and 1107 (1910); "The Oil Shales of the Maritime Provinces," by S. C. Ells, *J. Mining Soc. Nova Scotia*, 14 (1909–1910); "American Oil Shales," by Charles Baskerville and W. A. Hamor, *8th Intern. Cong. Appl. Chem.*, 25, 631 (1912); D. T. Day, "Mineral Resources of the United States," 11, 1071 (1913); "Oil-Shale of Northwestern Colorado and Northeastern Utah," by E. C. Woodruff and D. T. Day, Bull. No. 581-A, U. S. Geol. Survey, Wash., D. C. (1915); "Bituminous Shales of Colorado," by G. R. De Beque, *Eng. Mining J.*, 99, 773 (1915); "Oil Shale in Northwestern Colorado," by D. E. Winchester, Bull. No. 614-F, U. S. Geol. Survey, Wash., D. C. (1916); "The Bituminous Shale Industry in Northwestern Colorado," by G. R. De Beque, *Eng. Mining J.*, 102, 1011 (1916); "The Shale Oil Industry," by D. R. Steuart, *J. Soc. Chem. Ind.*, 14, 774 (1916); "Oil Resources of Black Shales of the Eastern U. S.," by G. H. Ashley, Bull. No. 641-L, U. S. Geol. Survey, Wash., D. C. (1917); "Oil Shales of the Uinta Basin, Northwestern Utah," by D. E. Winchester, Bull. No. 691-B. U. S. Geol. Survey, Wash., D. C. (Apr. 30, 1918); "Selected Bibliography on Oil Shale," by E. H. Burroughs and M. J. Gavin, Serial No. 2277, Bureau of Mines, Dept. Interior, Wash., D. C. (Sep., 1921); "Oil Shale: an Historical, Technical and Economic Study," by M. J. Gavin, Bull. No. 210, Bureau of Mines, Wash., D. C. (1924).

2 [p. 276] "The Thermal Decomposition of Shales," by R. H. McKee and E. E. Lyder, *J. Ind. Eng. Chem.*, 13, 613 (1921); **U. S. Pats.** 1280178 of Oct. 1, 1918 to D. T. Day; 1327572 of Jan. 6, 1920 to H. D. Ryan; 1643520 and 1643521 of Sep. 27, 1927 to C. S. Reeve; 1649545 of Nov. 15, 1927 to F. G. Renou; **Can. Pat.** 284743 of Nov. 13, 1928 to Australian Bituminous Compounds, Ltd.; **Brit. Pat.** 290886 of Sep. 20, 1927 to Australian Bituminous Compounds, Ltd.; **Ger. Pat.** 415222 of May 3, 1924 to H. W. Klever.

3 [p. 277] "Kerogen and Kerogen Shales," by E. H. Cunningham-Craig, *J. Inst. Petroleum Tech.*, 2, 238 (1916).

4 [p. 277] "Shaloilogy and Oil-Shale Nomenclature," by J. B. Jenson, *Chem. Met. Eng.*, 26, 509 (1922).

CHAPTER XIII

1 [p. 280] "Die Industrieteere, ihre Arten, Eigenschaften und Verwendung," by E. J. Fischer, *Teer u. Bitumen*, 27, 17, 33, 69 and 125 (1929); "Kunstliche Asphalte, Peche und ähnliche Produkte," by E. J. Fischer, *Teer u. Bitumen*, 28, 17 and 33 (1930); "Immediatepeche," by E. J. Fischer, *Teer u. Bitumen*, 31, 285 (1933); "Chemoasphalte und Chemopeche," by E. J. Fischer, *Teer u. Bitumen*, 33, 251 and 263 (1935).

2 [p. 280] **Ger. Pat.** 377353 of Mar. 9, 1918 to Walther Mathesius.

3 [p. 280] **U. S. Pat.** 1572552 of Feb. 9, 1926 to Joseph Michelman.

4 [p. 280] **Ger. Pats.** 424031 of Jul. 30, 1924 to Richard Levi; 455551 of Dec. 5, 1923 to Sinit A-G.

5 [p. 280] **U. S. Pat.** 1572510 of Feb. 9, 1926 to F. C. Atkinson.

6 [p. 280] **U. S. Pat.** 1603164 of Oct. 12, 1926 to W. R. Swint.

7 [p. 280] **Ger. Pats.** 13871 of Oct. 6, 1880 to E. Ernst; 15751 of Apr. 2, 1880 to Haring, Ehrenberg & Co. and M. Baswitz; 28838 of Mar. 18, 1884 to H. Propfe; 81341 of Aug. 28, 1894 to E. Meyer; 484000 of Dec. 9, 1924 to Les Nouvelles Industries Chimiques, S. A.

8 [p. 280] **Ger. Pat.** 349686 of Jun. 10, 1920 to Deutsche Gas A.-G.

9 [p. 282] "Coal Tar and Ammonia," by Georg Lunge, 5th Edition, D. Van Nostrand Co., New York (1916).

10 [p. 283] "Thermal Reactions of Aromatic Hydrocarbons in the Vapor Phase," by W. F. Rittman, O. Byron and Gustav Egloff, *J. Ind. Eng. Chem.*, 7, 1019 (1915); 8, 105 (1916); "The Pyrogenesis of Hydrocarbons," by A. E. Dunstan and F. B. Thole, *J. Ind. Eng. Chem.*, 9, 888 (1917).

CHAPTER XIV

1 [p. 288] "Wood Used for Distillation in 1906," Forest Service Circular No. 121, U. S. Dept. Agri., Wash., D. C. (Dec. 6, 1907); "Chemical Methods of Utilizing Wood," by F. P. Veitch, Circular No. 36, U. S. Dept. Agri., Wash., D. C. (Aug. 29, 1907); "Wood Distillation," by W. C. Geer, Forest Service Circular No. 14, U. S. Dept. Agri., Wash., D. C. (Nov. 5

1907); "Wood Turpentine, Its Production, Refining, Properties and Uses," Circular No. 144, U. S. Dept. Agri., Wash., D. C. (1912); "Yields from the Destructive Distillation of Certain Hardwoods," by J. F. Hawley and R. C. Palmer, Bull. No. 129, U. S. Dept. Agri., Wash., D. C. (Sep. 10, 1914); "Yields from the Destructive Distillation of Certain Hardwoods," by R. C. Palmer, Bull. No. 508, U. S. Dept. Agri., Wash., D. C. (Mar. 6, 1917); "The Distillation of Wood," by J. C. Lawrence, *J. Soc. Chem. Ind.*, **37**, 5T (1918); "The Influence of Moisture on the Yield of Products in the Destructive Distillation of Hardwood," by R. C. Palmer and H. Cloukey, *J. Ind. Eng. Chem.*, **10**, 262 (1918); "The Nomenclature of the Carbonization of Wood," by A. Bugge, *Z. angew. Chem.*, **39**, 1032 (1926); "Der Holzteer und seine technische Verwendung," by F. J. Fischer, *Teer*, **24**, 433 and 453 (1926).

2 [p. 289] "Yields from the Destructive Distillation of Certain Hardwoods," by R. C. Palmer, Table 5, p. 7, Bull. No. 508, U. S. Dept. Agri., Wash., D. C. (Mar. 6, 1917).

3 [p. 293] "Tar-still Operation in Hardwood Distillation Plants," by L. F. Hawley and H. N. Calderwood, Jr., *J. Ind. Eng. Chem.*, **12**, 684 (1920).

4 [p. 293] "Waste-wood Utilization by the Badger-Stafford Process," by W. G. Nelson, *J. Ind. Eng. Chem.*, **22**, 312 (1930).

5 [p. 295] **U. S. Pats.** 1108403 of Aug. 25, 1914 to S. E. Seaman; 1374887 of Apr. 12, 1921 to E. M. Sawtelle; 1375714 of Apr. 26, 1921 to H. N. McLeod; **Brit. Pats.** 119040 of Sep. 10, 1917 to O. F. Stafford; 131006 of Sep. 17, 1917 to Philip Poore; **Ger. Pat.** 387577 of Feb. 2, 1918 to Carbozit A.-G.

6 [p. 297] **Ger. Pat.** 336253 of May 9, 1920 to Gustav Ruth and Erich Usser.

7 [p. 299] "Wood-tar Distillation Residues," by Julius Marcusson and M. Picard, *Chem. Umschau*, **28**, 257 (1921).

8 [p. 300] "Wood-tar Distillation Residues," by Julius Marcusson and M. Picard, *Chem. Umschau*, **28**, 257 (1921); "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, Verlag von Wilhelm Engelman, Leipzig, p. 72 (1921).

9 [p. 300] "The Free Carbon of Wood-tar Pitches," by H. K. Benson and L. L. Davis, *J. Ind. Eng. Chem.*, **9**, 141 (1917).

10 [p. 300] "Examination of Asphalts," by Edward Donath and B. M. Margosches, *Chem. Ind.*, **27**, 220 (1904); "Behavior of Wood-tar Pitch with Certain Organic Solvents," by B. M. Margosches, *Chem. Rev. Fett- u. Harz-Ind.*, **12**, 5 (1905); "Distinction between Lignite Pitch and Other Pitches," by Edmund Graefe, *Chem. Zeit.*, **30**, 298 (1906).

11 [p. 301] **U. S. Pat.** 1912435 of Jun. 6, 1933 to F. M. Damitz.

12 [p. 301] **U. S. Pat.** 1933796 of Nov. 7, 1933 to L. J. Figg, Jr.

13 [p. 301] **U. S. Pat.** 2008979 of Jul. 23, 1935 to L. J. Figg, Jr.; **Ger. Pat.** 286650 of Jul. 17, 1914 to Chem. Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.

14 [p. 301] **Ger. Pat.** 286650 of Jul. 17, 1914 to Chem. Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.

15 [p. 301] **Ger. Pats.** 338854 of Dec. 11, 1919 to Chem. Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.; 406150 of Mar. 25, 1923 to Rütgerswerke A.-G.; **Can. Pat.** 331165 of Mar. 21, 1933 to Tennessee Products Corp.

16 [p. 301] **Ger. Pat.** 324876 of Dec. 12, 1917 to Raszbach & Kralle, and J. Altendorf.

17 [p. 301] **Ger. Pat.** 320620 of Jul. 11, 1918 to J. D. Riedel A.-G.

18 [p. 301] **U. S. Pats.** 82419 of Sep. 22, 1868 to J. L. Kidwell; 540792 of Jun. 11, 1895 to J. A. Just.

19 [p. 301] **U. S. Pat.** 1841235 of Jan. 12, 1932 to H. A. Winkelmann.

20 [p. 301] **Ger. Pats.** 163446 of Jun. 18, 1903 to Chem. Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.; 171379 of Jan. 15, 1904 to Chem. Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.; 332634 of Nov. 5, 1918 to Alexander Flexer.

21 [p. 301] "Wood Tar and Its Technical Application," by E. J. Fischer, *Teer und Bitumen*, **24**, 433, 453, 601 and 619 (1926).

22 [p. 302] Bul. No. 229, U. S. Dept. Agri., Wash, D. C. (Jul. 28, 1915).

23 [p. 303] "The Distillation of Resins," by Victor Schweitzer, p. 59, D. Van Nostrand Co., New York (1906).

24 [p. 304] "Artificial Asphalt," by E. Valenta, Zentralorgan für Warenkunde und Technologie, **1**, 19 (1891); *Chem.-Zeit. Rep.*, **15**, 211 (1891).

CHAPTER XV

1 [p. 305] "Peat," by E. Ries, 55th Annual Rept. N. Y. State Museum, p. 55 (1903); "Peat Fuel: its Manufacture and Uses," by W. E. H. Carter, Report Ontario Bureau of Mines for 1903, Toronto, Can. (1904); "Die Moore der Schweiz," by J. Früh and C. Schroeter, Beitr. Geol. Schweiz Geotech., Series III, Bern (1904); "Canadian Peat," by R. Chalmers, Mineral Res. Canada (1904); "Peat Deposits of Northern New Jersey," by W. E. McCourt and C. W. Parmelee, Ann. Rept. State Geologist of N. J. (1905); "Peat," by A. L. Parsons, 57th Annual Rept. N. Y. State Museum, Vol. I, p. 16 (1905); "Peat," by J. A. Holmes, Bull. No. 200, U. S. Geol. Survey, pp. 11–15, Wash., D. C. (1906); "Peat," Annual Rept. New Jersey State Geologist for 1905, Trenton, N. J. (1906); "The Data of Geochemistry," by F. W. Clarke, Bull. No. 330, U. S. Geol. Survey, Wash., D. C. (1908); "Peat and Lignite, Their Manufacture and Uses in Europe," by E. Nystrom, Bull. No. 19, Canada Dept. of Mines, Ottawa, Can. (1908); "Investigation of the Peat Bogs and Peat Industry of Canada," by E. Nystrom and S. A. Anrep, Report No. 30, Canada Dept. of Mines, Ottawa, Can. (1908); "Reports on the Irish Peat Industries," by Hugh Ryan, Econ. Proc. Roy. Soc. Dublin, Vol. I, Parts 10 and 13 (1907–8); "The Utilization of Peat for Making Gas or Charcoal with Recovery of By-products," by H. R. Sankey, Engineering (Sep. 11, 1908); "Investigation of the Peat Bogs and Peat Industry of Canada," Report No. 71, by A. Anrep, Jr., Larson, Ekelund, etc., Ottawa, Can. (1909–1910); Report No. 266, by A. Anrep, Jr. (1911–1912); Report No. 351, by A. Anrep, Jr. (1913–1914); Canada Dept. of Mines, Ottawa, Can.; "The Uses of Peat," by Chas. A. Davis, Bull. No. 16, Bureau of Mines, Dept. of Interior, Wash., D. C. (1911).

2 [p. 306] "Zur Analyse des Torfes," by H. Bornträger, *Z. anal. Chem.*, **39**, 694 (1900); **40**, 639 (1901).

3 [p. 307] "Use of Peat in Gas Producing Plants," by E. C. C. Baly, *J. Soc. Chem. Ind.*, **35**, 1240 (1916).

4 [p. 310] "Peat and Lignite, Their Manufacture and Uses in Europe," by E. Nystrom, Bull. No. 19, Canada Dept. of Mines, Ottawa, Can. (1908); "Peat, Lignite and Coal," by B. F. Haanel, Report No. 299, Canada Dept. of Mines, Ottawa, Can. (1913); "The Production and Use of Browncoal in the Vicinity of Cologne, Germany," by C. A. Davis, Tech. Paper No. 55, Bureau of Mines, Wash., D. C. (1913); "The Uses of Lignite," by C. A. Davis, Tech. Paper No. 55, Bureau of Mines, Wash., D. C. (1913); "Methods of Utilizing Lignite," by S. M. Darling, *J. Gas Lighting*, **131**, 456 (1915); "The Investigation of Six Samples of Alberta Lignites," by B. F. Haanel and J. Blizzard, Report No. 331, Dept. of Mines, Ottawa, Can. (1915); "The Brown Coal Distillation Industry of Germany," by D. R. Steuart, *J. Soc. Chem. Ind.*, **36**, 167 (1917); "Ueber die Aufarbeitung von Braunkohlenteer und Schieferöl," by Erich Koch, *Teer*, **25**, 65 (1927).

5 [p. 313] "Low-temperature Brown-coal Tar," by R. Schmidt, *Brennstoff-Chem.*, **16**, 241 (1935).

6 [p. 313] "Briquetting Tests of Lignite at Pittsburgh, Pa.," Bull. No. 14, Bureau of Mines, U. S. Dept. of Interior, Wash., D. C. (1911).

7 [p. 318] "The Composition of Lignite Tars," by Julius Marcusson and M. Picard, *Mitt. Materialprüfungsamt*, **39**, 329 (1921).

8 [p. 319] **Ger. Pat.** 333061 of Oct. 24, 1918 to Deutsche Erdöl A.-G.

9 [p. 322] **Hungarian Pat.** 105143 of Feb. 15, 1933 to László Bregár.

10 [p. 323] "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson; p. 60, Wilhelm Engelmann, Leipzig (1921).

11 [p. 323] "Distinction between Lignite Pitch and Other Pitches," by Edmund Graefe, *Chem. Zeit.*, **30**, 298 (1906); "Native and Artificial Asphalts," by Julius Marcusson and R. Eickmann, *Chem. Rev. Fett- u. Harz-Ind.*, **15**, 315 (1908); "Identifying Asphalts," by Julius Marcusson, *Chem. Rev. Fett- u. Harz-Ind.*, **18**, 47 (1911); *J. Soc. Chem. Ind.*, **30**, 480 (1911); "Chemical Composition and Examination of Natural and Artificial Asphalts," by Julius Marcusson, *Chem. Rev. Fett- u. Harz-Ind.*, **19**, 166 (1912).

12 [p. 323] **Ger. Pat.** 335748 of Jan. 30, 1920 to Arthur Fürth and Helene Japhé.

13 [p. 323] **Ger. Pat.** 431742 of Jul. 8, 1924 to Gesellschaft für Gas- u. Teerverwertung, m.b.H.

14 [p. 323] **Austrian Pat.** 104402 of May 15, 1926 to Josef Drescher.
15 [p. 323] **Brit. Pat.** 331846 of Apr. 4, 1929 to Frankfurter Gas-Gesellschaft and H. W. Hoelzer.

CHAPTER XV

1 [p. 329] "Oil-Shale: an Historical, Technical and Economic Study," by M. J. Gavin, Bull. No. 210, Bureau of Mines, Denver, Colorado (1922).
2 [p. 330] "The American Petroleum Industry," by R. F. Bacon and W. A. Hamor, Vol. II, p. 832, McGraw-Hill Book Co., Inc., New York (1916); "Oil Resources of Black Shales of the Eastern United States," by G. H. Ashley, Bull. No. 641-L, U. S. Geol. Survey, Wash., D. C. (1917).
3 [p. 335] "Mixtures of Coal Tar and Estobitumen," by K. Luts, *Asphalt u. Teer*, 31, 774 (1931); "Ueber die Untersuchung von estnischen Brennschieferasphalten unter Anwendung einiger neuartiger Verfahren," by K. A. Hoepfner, *Asphalt u. Teer Strassenbautechnik*, 32, 777, 791, 807, 823, 835 and 847 (1932).

CHAPTER XVII

1 [p. 336] "Coal-tar Products," by Horace C. Porter and C. G. Storm. Circ. No. 89, Bureau of Mines, Dept. of Interior, Wash., D. C. (1916); "Preparations and Uses of Tar and Its Simple Crude Derivatives," by W. W. Odell, Technical Paper No. 268, Bureau of Mines, Dept. of Interior, Wash., D. C. (1922).
2 [p. 344] "Coke-oven Tars of the United States," by Prévost Hubbard, Circ. No. 97, Office of Public Roads, U. S. Dept. of Agri., Wash., D. C. (Feb. 7, 1912); "By-Products Recovered in the Manufacture of Coke," by W. H. Childs, Am. Iron and Steel Inst., New York (May 26, 1916).
3 [p. 344] **U. S. Pat.** 36632 of Oct. 7, 1862 to A. H. Perkins.
4 [p. 346] "Some of the Constituents of Coke-oven Tar," by J. M. Weiss and C. R. Downs, *J. Ind. Eng. Chem.*, 15, 1022 (1923).
5 [p. 347] "Cleaning of Blast-furnace Gases," by F. H. Wagner, 1st Edition, McGraw-Hill Book Co., New York (1914); "Blast-furnace Construction in America," by J. E. Johnson, Jr., London (1917).
6 [p. 348] "Producer-gas Development in Europe," by R. H. Fernald, Bull. No. 4, Bureau of Mines, Wash., D. C. (1911); "Gas Engines and Producers," by L. S. Marks and H. S. McDewell, Chicago, (1916); "The Tars Distilled from Bituminous Coal in Hand-fired Furnaces," by H. S. Katz, Tech. Paper No. 195, Bureau of Mines, Dept. of Interior, Wash., D. C. (1918).
7 [p. 348] "Chemistry of Low-temperature Tar," by G. T. Morgan, *J. Soc. Chem. Ind.*, 51, 67T (1932).
8 [p. 350] "New Reaction of Low-temperature Tars," by F. Schulz and J. Prunet, Bull. Soc. Chim., 35, 638 (1924).
9 [p. 353] **Brit. Pat.** of 1912 (Jul. 13), 16385 to H. P. Hird, Norwood Green, E. V. Chambers and T. C. Hammond.
10 [p. 353] **U. S. Pat.** 2064486 of Dec. 15, 1936 to S. P. Miller; **Can. Pat.** 309218 of Mar. 10, 1931 to Barrett Co.; **Brit. Pat.** 127700 of May 28, 1918 to T. O. Wilton.
11 [p. 353] **Brit. Pat.** 123163 of Feb. 14, 1919 to William Blakeley.
12 [p. 353] "Tar Distillation in the U. S.—General Development and Recent Progress," R. P. Perry, *8th Intern. Cong. Applied Chem.*, 10, 242 (1912); "Tar Refining and Tar Products," by J. M. Weiss, Am. Gas Assoc., Chap. VIII, New York (1926).
13 [p. 354] "Tar Distillation in the United States," by R. P. Perry, *8th Intern. Cong. Applied Chem.*, 10, 233 (1912); "Coal-tar Light Oil in the United States," by J. M. Weiss, *8th Intern. Cong. Applied Chem.*, 10, 287 (1912); "Coal Tar and Ammonia," by Georg Lunge, Guiney-Jackson, London (1915); "Coal Tar Disposal," by J. M. Weiss, Proc. Intern. Conference on Bituminous Coal (Nov. 15th to 18th 1926); "Tar Refining and Tar Products," Am. Gas Assoc., Chap. VIII, by J. M. Weiss (1926).

14 [p. 360] **U. S. Pat.** 1821144 of Sep. 1, 1931 to J. V. E. Dickson.

15 [p. 350] **U. S. Pat.** 1814989 of Jul. 14, 1931 to Carl Wessel.

16 [p. 351] "The Distillation of Coal Tar," by J. M. Weiss, *J. Soc. Chem. Ind.*, **51**, 219 and 246 (1932).

17 [p. 361] **U. S. Pats.** 1418893 of Jun. 6, 1922 to J. M. Weiss; 2007656 of Jul. 9, 1935 to J. V. Meigs; 2074689 of Mar. 23, 1937 to G. T. Gambrill, Jr.; **Brit. Pat.** 158852 of Apr. 1, 1920 to Barrett Co.; **Ger. Pat.** Appl. G-82798 of Jun. 2, 1932 to Gewerkschaft Mathias Stinnes.

18 [p. 362] **U. S. Pats.** 1214259 of Jan. 30, 1917 to Johann Artmann; 1415056 and 1415057 of May 9, 1922 to Charles Schaer; **Brit. Pat.** 104636 of Mar. 15, 1917 to Harold Moore and C. Day; **Ger. Pats.** 221898 of Feb. 19, 1907 to A. Reichman; 260060 of Oct. 27. 1912 to Friedrich Raschig; 268063 of Feb. 9, 1913 to Gebrüder Keller.

19 [p. 362] **Brit. Pat.** of 1912 (May 2), 11143 to H. P. Hird.

20 [p. 363] "Continuous Tar Distillation in Koppers Pipe-Stills," by O. Eisler, Z. Zamrzla and M. Weinkopf, *Glückauf*, **72**, 184 (1936); **Brit. Pat.** 127700 of May 28, 1919 to T. O. Wilton.

21 [p. 363] **Ger. Pat.** 238013 of Nov. 6, 1910 to A. Lemmer.

22 [p. 363] **Ger. Pat.** 292992 of May 23, 1915 to B. Leinweber.

23 [p. 363] **U. S. Pats.** 1821144 of Sep. 1, 1931 to J. V. E. Dickson; 2005077 of Jun. 18, 1935 to J. V. E. Dickson; **Can. Pat.** 348280 of Feb. 19, 1935 to Barrett Co.; **Brit. Pats.** 351388 and 351389 of Jan. 17, 1930 to Barrett Co.; 345654 of Nov. 30, 1928 to Barrett Co.; 346407 of Dec. 15, 1928 to Barrett Co.

24 [p. 363] **U. S. Pat.** 2029883 of Feb. 4, 1936 to A. A. McCubbin and Joseph Zavertnik, Jr.

25 [p. 364] "The Direct Recovery of Standard Road Tars and Other Tar Constituents of Coal Distillation Gases by Fractional Condensation," by F. Cooke, *Gas World*, **92** (Coking Section), 13 (1930); "Coal-tar Distillation by Hot Gas at the Coke-oven Plant," by G. E. McCloskey, *Chem. Met. Eng.*, **39**, 333 (1932); **U. S. Pats.** 1826428, 1826429, 1826430 and 1826431 of Oct. 6, 1931 to S. P. Miller; 1844741 of Feb. 9, 1932 to W. M. Bywater; 1844889, 1844890, 1844891, 1844892, 1844893, 1844894, 1844895, 1844896, 1844897, 1844898, 1844899, 1844900 and 1844901 of Feb. 9, 1932 to S. P. Miller; 1849197, 1849198 and 1849199 of Mar. 15, 1932 to S. P. Miller; 1862493 and 1862494 of Jun. 7, 1932 to S. P. Miller; 1868394 and 1868395 of Jul. 19, 1932 to S. P. Miller; 1868470 of Jul. 19, 1932 to L. R. Forrest and E. B. Conklin; 1871550 of Aug. 16, 1932 to S. P. Miller; 1871596 of Aug. 16, 1932 to E. H. Ellms; 1871628 of Aug. 16, 1932 to G. E. McCloskey and W. B. Wingert; 1876594 of Sep. 13, 1932 to Joseph Becker; 1883642 of Oct. 18, 1932 to E. H. Ellms; 1884084, 1884085, 1884086 and 1884087 of Oct. 25, 1932 to S. P. Miller; 1884220 of Oct. 25, 1932 to A. H. Radasch; 1886967 of Nov. 8, 1932 to S. P. Miller; 1888235 of Nov. 22, 1932 to S. P. Miller; 1892469 of Dec. 27, 1932 to S. P. Miller; 1892654 of Dec. 27, 1932 to S. P. Miller; 1893145 of Jan. 3, 1933 to S. P. Miller; 1894471 of Jan. 17, 1933 to G. E. McCloskey; 1895676 of Jan. 31, 1933 to S. P. Miller; 1898980 of Feb. 21, 1933 to S. P. Miller; 1904161 of Mar. 18, 1933 to S. P. Miller; 1904504 and 1904505 of Apr. 18, 1933 to S. P. Miller; 1907078 of May 2, 1933 to G. E. McCloskey; 1909978 of May 23, 1933 to S. P. Miller; 1912294 of May 30, 1933 to S. P. Miller; 1912393 of Jun. 6, 1933 to Fred Osborne; 1912394 of Jun. 6, 1933 to S. P. Miller; 1915251 of Jun. 20, 1933 to S. P. Miller; 1918709 of Jul. 18, 1933 to S. P. Miller; 1919240 and 1919241 of Jul. 25, 1933 to S. P. Miller; 1919704 of Jul. 25, 1933 to Fred Osborne; 1920097 of Jul. 25, 1933 to S. P. Miller; 1920163 of Jul. 25, 1933 to S. P. Miller; 1920267 of Aug. 1, 1933 to S. P. Miller; 1920801 of Aug. 1, 1933 to S. P. Miller; 1921300 of Aug. 8, 1933 to S. P. Miller; 1924163 of Aug. 29, 1933 to S. P. Miller; 1925150 of Sep. 5, 1933 to S. P. Miller; 1927280 of Sep. 19, 1933 to S. P. Miller; 1927298 of Sep. 19, 1933 to A. H. Radasch; 1930124 of Oct. 10, 1933 to L. R. Forrest; 1930130 of Oct. 10, 1933 to S. P. Miller; 1935326 of Nov. 14, 1933 to S. P. Miller; 1942195 of Jan. 2, 1934 to J. M. Weiss; 1942371, 1942372, 1942373, 1942374 and 1942375 of Jan. 2, 1934 to S. P. Miller; 1944129, 1944130 and 1944131 of Jan. 16, 1934 to S. P. Miller; 1944523 of Jan. 23, 1934 to S. P. Miller; 1947485 of Feb. 20, 1934 to S. P. Miller; 1952020 of Mar. 20, 1934 to S. P. Miller; 1958277 and 1958278 of May 8, 1934 to S. P. Miller; 1958416 of May 15, 1934 to G. E. Brandon; 1958440 of May 15, 1934 to S. P. Miller; 1958450

of May 15, 1934 to C. G. Strupp; 1958583, 1958584, 1958585 and 1958586 of May 15, 1934 to S. P. Miller; 1958849 of May 15, 1934 to E. H. Ellms; 1959290 of May 15, 1934 to S. P. Miller; 1969472 of Aug. 7, 1934 to S. P. Miller; 1972468 of Sep. 4, 1934 to Joseph Zavertnik, Jr.; 1976243 of Oct. 9, 1934 to S. P. Miller; 1979046 of Oct. 30, 1934 to S. P. Miller; 1979838 of Nov. 6, 1934 to G. E. McCloskey; 1983915 of Dec. 11, 1934 to G. E. McCloskey; 2002704 of May 28, 1935 to S. P. Miller; 2005102 of Jun. 18, 1935 to S. P. Miller; 2007378 of Jul. 9, 1935 to S. P. Miller; 2018377 of Oct. 22, 1935 to S. P. Miller; 2040096, 2040097, 2040098 and 2040099 of May 12, 1936 to S. P. Miller; 2040100 and 2040101 of May 12, 1936 to S. P. Miller; 2040104 of May 12, 1936 to A. H. Radasch; 2044164 of Jun. 23, 1936 to W. M. Bywater; 2045407 of Jun. 23, 1936 to A. H. Radasch; 2067450 of Jan. 12, 1937 to H. E. Imes; 2068304 of Jan. 19, 1937 to E. O. Rhodes; **Can. Pats.** 285502 of Dec. 11, 1928 to Barrett Co.; 288241 of Mar. 26, 1929 to Barrett Co.; 289618 of May 14, 1929 to Barrett Co.; 289794 and 289796 of May 21, 1929 to Barrett Co.; 295737, 295738 and 295739 of Dec. 17, 1929 to Barrett Co.; 298816 of Apr. 1, 1930 to Barrett Co.; 299528 and 299529 of Apr. 22, 1930 to Barrett Co.; 300539 and 300540 of May 27, 1930 to Barrett Co.; 304940, 304941, 304942, 304943, 304944, 304945 and 304946 of Oct. 21, 1930 to Barrett Co.; 306727 and 306728 of Dec. 16, 1930 to Barrett Co.; 310823 and 310824 of Apr. 28, 1931 to Barrett Co.; 311252, 311253, 311254, 311255, 311256, 311257, 311258 and 311259 of May 12, 1931 to Barrett Co.; 311674 of May 26, 1931 to Barrett Co.; 313014, 313015, 313016, 313017, 313018, 313019, 313020 and 313021 of Jul. 7, 1931 to Barrett Co.; 316597 of Nov. 3, 1931 to W. M. Bywater; 316606 of Nov. 3, 1931 to E. H. Ellms; 316638 of Nov. 3, 1931 to S. P. Miller; 316644 of Nov. 3, 1931 to G. E. McCloskey; 316684 and 316685 of Nov. 3, 1931 to Barrett Co.; 320350 of Mar. 8, 1932 to Barrett Co.; 320758 of Mar. 22, 1932 to Barrett Co.; 324500 of Jul. 26, 1932 to Barrett Co.; 345315 of Oct. 16, 1934 to Barrett Co.; 363129, 363130 and 363131 of Jan. 5, 1937 to Barrett Co.; **Brit. Pats.** 282367 of Nov. 8, 1927 to Barrett Co.; 282826 of Dec. 30, 1927 to Barrett Co.; 284703 of Jan. 28, 1928 to Barrett Co.; 285907 of Feb. 26, 1927 to Barrett Co.; 286278 of Mar. 2, 1927 to Barrett Co.; 287084 of Mar. 14, 1927 to Barrett Co.; 287900 of Mar. 21, 1927 to Barrett Co.; 288270 of Apr. 6, 1927 to Barrett Co.; 289023 of Apr. 20, 1927 to Barrett Co.; 289378 of Apr. 25, 1927 to Barrett Co.; 289832 of May 3, 1927 to Barrett Co.; 294106 of Jul. 6, 1927 to Barrett Co.; 295687 of Aug. 17, 1927 to Barrett Co.; 298153 of Oct. 4, 1927 to Barrett Co.; 303093 of Feb. 14, 1928 to Barrett Co.; 303163, 303164, 303165, 303166 and 303167 of Dec. 27, 1928 to Barrett Co.; 306093 of Feb. 15, 1928 to Barrett Co.; 308577 of Mar. 23, 1928 to Barrett Co.; 309946 of Feb. 16, 1929 to Barrett Co.; 315368 of Mar. 2, 1928 to Barrett Co.; 315373 and 315374 of Mar. 2, 1928 to Barrett Co.; 345272 of Dec. 18, 1929 to Barrett Co.; 345654 of Nov. 21, 1929 to Barrett Co.; 346753 of Jan. 10, 1930 to Barrett Co.; 346805 of Dec. 18, 1929 to Barrett Co.; 347107 of Jan. 16, 1929 to Barrett Co.; 347240 of Dec. 18, 1928 to Barrett Co.; 349088 of Dec. 18, 1928 to Barrett Co.; 351388 and 351389 of Jan. 26, 1929 to Barrett Co.; 354591 of Sep. 18, 1930 to Barrett Co.; 355031 of Aug. 20, 1930 to Barrett Co.; 357885 of Dec. 22, 1930 to Barrett Co.; 358233 of Sep. 13, 1930 to Barrett Co.; 358375 of Dec. 22, 1930 to Barrett Co.; 367842 of Aug. 22, 1930 to Barrett Co.; 370387 of Dec. 3, 1930 to Barrett Co.; **French Pats.** 686723, 686724, 686725 and 686726 of Dec. 16, 1929 to Barrett Co.; 687890 of Jan. 9, 1930 to Barrett Co.; 688298 of Jan. 15, 1930 to Barrett Co.; 688837 and 688838 of Jan. 25, 1930 to Barrett Co.

26 [p. 366] "Coal-tar and Water-gas Tar Creosotes—Their Properties and Methods of Testing," by Ernest Bateman, Bull. No. 1036, U. S. Dept. of Agri., Wash., D. C. (1922).

27 [p. 367] "Pechverladeeinrichtigungen," by Fr. Bürk, *Teer*, **24**, 109 (1926); **Ger. Pats.** 272140 of Aug. 7, 1913 to F. C. Still; 409727 of Feb. 1, 1924 to O. Friese.

28 [p. 367] **U. S. Pats.** 1088635 of Feb. 24, 1914 to C. N. Stevens; 1327354 of Jan. 6, 1920 to R. P. Perry.

29 [p. 367] **U. S. Pats.** 1088635 of Feb. 24, 1914 to C. N. Stevens; 1210097 of Dec. 26, 1916 to R. P. Perry and E. F. Stewart; 1807223 of May 26, 1931 to S. P. Miller.

30 [p. 367] **U. S. Pats.** 1289892 of Dec. 31, 1918 to R. P. Perry; 1402177 of Jan. 3, 1922 to R. P. Perry.

31 [p. 367] **U. S. Pats.** 1088635 of Feb. 24, 1914 to C. N. Stevens; 1962515 of Jun. 12, 1934 to S. P. Miller; **Ger. Pat.** 286549 of Nov. 8, 1913 to Barrett Mfg. Co.

32 [p. 367] **Brit. Pat.** 162727 of Jan. 2, 1920 to F. J. Commin.

33 [p. 367] **U. S. Pats.** 1946446 of Feb. 6, 1934 to E. O. Rhodes and E. H. Hyde; 1976908 of Oct. 16, 1934 to Lester Wittenberg; 2030528 of Feb. 11, 1936 to S. P. Miller; 2030575 of Feb. 11, 1936 to E. H. Ellms; 2070961 of Feb. 16, 1937 to C. S. Reeve; **Can. Pat.** 352524 of Aug. 20, 1935 to Shell Development Co.

34 [p. 367] **U. S. Pat.** 2007378 of Jul. 9, 1935 to S. P. Miller; 2033885 of Mar. 10, 1936 to I. H. Derby; **Can. Pat.** 327612 of Nov. 15, 1932 to Barrett Co.; **Brit. Pat.** 351023 of Mar. 19, 1930 to Thermal Industrial & Chem. (T. I. C.); Res. Co., Ltd. and C. O. Condrup; **Ger. Pat.** 510940 of Dec. 25, 1928 to Frank Cooke.

35 [p. 367] **U. S. Pat.** 1917384 of Jul. 11, 1933 to G. E. McCloskey.

36 [p. 368] **Brit. Pat.** 384761 of Nov. 5, 1931 to T. Cheeseman.

37 [p. 368] **Ger. Pat.** 420394 of Sep. 5, 1923 to Z. M. Stinnes and Anton Weindel.

38 [p. 368] **U. S. Pat.** 1853699 of Apr. 12, 1932 to Emil Rahmann.

39 [p. 368] **U. S. Pats.** 2033545 of Mar. 10, 1936 to A. W. Ralston, C. W. Christensen and S. T. Bauer; 2033547 of Mar. 10, 1936 to A. W. Ralston.

40 [p. 368] **U. S. Pats.** 1743433 of Jan. 14, 1930 to A. B. Cowdery; 1820444 of Aug. 25, 1931 to A. B. Cowdery; **Can. Pat.** 319932 of Feb. 23, 1932 to Barrett Co.

41 [p. 368] **U. S. Pats.** 1957468 and 1957469 of May 8, 1934 to S. P. Miller and M. L. Hamlin.

42 [p. 369] **U. S. Pat.** 1355103 of Oct. 5, 1920 to G. C. Bailey; 2066386 of Jan. 5, 1937 to F. H. Bergeim.

43 [p. 369] **U. S. Pat.** 1853699 of Apr. 12, 1932 to Emil Rahmann.

44 [p. 369] **U. S. Pat.** 1782341 of Nov. 18, 1930 to S. I. Charlesworth and A. W. Harris.

45 [p. 370] **U. S. Pat.** 1511192 of Oct. 7, 1924 to R. P. Perry; **Brit. Pat.** 155226 of Aug. 10, 1920 to R. P. Perry.

46 [p. 370] **U. S. Pat.** 2064484 of Dec. 15, 1936 to J. V. Meigs; **Brit. Pat.** 188307 of Sep. 4, 1922 to T. H. Hall.

47 [p. 370] **U. S. Pat.** 1230782 of Jun. 19, 1917 to P. C. Reilly.

48 [p. 370] **U. S. Pat.** 1884339 of Oct. 25, 1932 to F. W. Steere.

49 [p. 370] **U. S. Pat.** 1942980 of Jan. 9, 1934 to E. O. Rhodes.

50 [p. 370] **U. S. Pat.** 1813341 of Jul. 7, 1931 to O. D. Cunningham.

51 [p. 370] "Some Experiments on Technical Bitumens," by S. R. Church and J. M. Weiss, *Proc. A. S. T. M.*, **15**, Part II, 274 (1915).

52 [p. 373] "Chemische Zusammensetzung und Untersuchung der natürlichen und künstlichen Asphalte," by Julius Marcusson, *Chem. Rev. Fett-u. Harz-Ind.*, **19**, 166 (1912); "Nachweis von Naturasphalt und Erdölpech in Rückständen der Steinkohlenteerdestillation," by F. Schwartz, *Chem. Rev. Fett- u. Harz-Ind.*, **20**, 28 (1913).

53 [p. 374] **U. S. Pat.** 298072 of May 6, 1884 to D. H. Dorsett; **Brit. Pat.** 163199 of Apr. 20, 1920 to H. Tindale; **Ger. Pat.** 335748 of Jan. 30, 1920 to Arthur Fürth and Helene Japhé; **French Pat.** 639838 of Aug. 18, 1927 to Alexander Flexer.

54 [p. 374] **U. S. Pat.** 298072 of May 6, 1884 to D. H. Dorsett.

55 [p. 374] **U. S. Pats.** 1155443 of Oct. 5, 1915 to Jean Rosen; 1259674 of Mar. 19, 1918 to Jean Rosen; 1273927 of Jul. 30, 1918 to Jean Rosen; **Brit. Pat.** 447861 of Jul. 23, 1935 to H. D. Elkington; **Ger. Pat.** 626513 of Feb. 27, 1936 to Wigankow & Sachse Neuzeitlicher Strassenbau, G.m.b.H.

56 [p. 374] **U. S. Pat.** 1044175 of Nov. 12, 1912 to Henri Hennebutte.

57 [p. 374] **Ger. Pats.** 383922 of Jan. 20, 1922 to Rütgerswerke A.-G.; 550151 of Jun. 8, 1927 to Stadtgemeinde Dresden.

58 [p. 374] **Ger. Pat.** Appl. E-43419 of Oct. 19, 1932 to Enterprises Albert Cochery, S. A.

59 [p. 374] **Brit. Pat.** 433248 of Oct. 19, 1934 to Lonza Elektrizitätswerke und Chem. Fabrik A.-G.

60 [p. 374] **Brit. Pat.** 397484 of Dec. 23, 1931 to O. Reynard and E. H. Thompson.

61 [p. 374] **Ger. Pats.** Appl. J-48234 of Oct. 30, 1933 to Felix Jacobsohn; 626973 of Mar. 7, 1936 to Felix Jacobsohn; 627606 of Nov. 1, 1933 to Felix Jacobsohn.

62 [p. 374] **Brit. Pat.** 149979 of Aug. 10, 1920 to Franz Fischer; **Ger. Pat.** 335748 of Jan. 30, 1920 to Arthur Fürth and Helene Japhé.

63 [p. 374] **Ger. Pats.** 163446 of Jun. 18, 1903 to Chemische Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.; 171380 of Jan. 26, 1904 to Chemische Fabrik Flörsheim and Dr. H. Noerdlinger A.-G.; 367495 of Dec. 21, 1920 to Rütgerswerke A.-G.; 603762 of Mar. 7, 1933 to Gewerkschaft Mathias Stinnes; Appl. D–68637 of Aug. 23, 1934 to Karl Drees and Georg Kowalski; **Austrian Pat.** 110401 of Mar. 15, 1928 to Albert Turek.

64 [p. 374] **U. S. Pats.** 746688 and 746689 of Dec. 15, 1903 to D. H. Dupont-Franklin; **Brit. Pats.** of 1903 (Jun. 23), 14011 to C. E. Pensa; of 1903 (Oct. 23), 22986 to L. L. A. Seguin and J. F. G. de Roussy.

65 [p. 374] **French Pat.** 593730 of Feb. 21, 1925 to K. V. Nielsen.

66 [p. 374] **U. S. Pat.** 903287 of Nov. 10, 1908 to H. V. D. Heide.

67 [p. 374] **Ger. Pat.** 132661 or Feb. 24, 1901 to Eduard Oehring.

68 [p. 374] **Ger. Pat.** 431742 of Jul. 8, 1924 to Ges. für Gas- und Teerverwertung m.b.H.

69 [p. 375] **U. S. Pats.** 39139 of Jul. 7, 1863 to J. G. Fuller; 59462 of Nov. 6, 1866 to H. K. Schauck; 60830 of Jan. 1, 1867 to J. F. Boynton; 63367 of Apr. 2, 1867 to Joseph Darby; 74083 of Feb. 4, 1868 to David Harger; 82419 of Sep. 22, 1868 to J. L. Kidwell; 101970 of Apr. 19, 1870 to M. G. Balfour; 149077 of Mar. 31, 1874 to Gottlob Steinemann; Reissue 5976 of Jul. 21, 1874 to Gottlob Steinemann; 165530 of Jul. 13, 1875 to H. R. Bellamy; 171720 of Jan. 4, 1876 to T. H. Dunham; 185050 of Dec. 5, 1876 to H. K. Schanck; 187748 of Feb. 27, 1877 to J. C. Cheatham; 210406 and 210411 of Dec. 3, 1878 to A. G. Day; 256368 of Apr. 11, 1882 to G. H. Pöschel; 321956 of Jul. 14, 1885 to J. W. Ellis; 403548 of May 21, 1889 to B. E. Olsen and Charles Gabriel; 540792 and 540793 of Jun. 11, 1895 to J. A. Just; 598147 of Feb. 1, 1898 to Albert Hannemann; 624081 and 624082 of May 2, 1899 to D. W. Peck; 1266261 of May 14, 1918 to G. A. Henderson; 1353003 of Sep. 14, 1920 to J. J. White, Jr.; **Can. Pats.** 49753 of Aug. 24, 1895 to J. A. Just; 54327 of Dec. 11, 1896 to J. A. Just; **Brit. Pats.** 151852 of Oct. 7, 1920 to Chikakiyo Honda; 291749 of Jun. 7, 1928 to W. Schroth; 306421 of Feb. 18, 1929 to Fritz Hofmann and Robert Uloth; **Ger. Pats.** 61555 of May 21, 1891 to Georg Friedrich & Co.; 65239 of Nov. 10, 1891 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 122387 of Nov. 20, 1900 to Albin Karg; 124629 of Apr. 29, 1899 to D. W. Peck; 143147 of Feb. 27, 1901 to Clara Hermes; 226519 of Aug. 16, 1908 to Eduard Dyckerhoff; 226520 of Feb. 15, 1910 to Eduard Dyckerhoff; 236239 of Mar. 11, 1910 to Société Cuel, Pinguet et Cie.; 330970 of Jun. 24, 1919 to Verkaufsvereiningung für Teererzeugnisse G.m.b.H. and Fritz Schreiber; 332888 of Aug. 12, 1919 to Verkaufsvereinigung für Teererzeugnisse G.m.b.H.; 425126 and 425127 of Dec. 28, 1924 to Karl Rösler.

70 [p. 375] **Can. Pat.** 13109 of Jul. 16, 1881 to Paul Crippen.

71 [p. 375] **U. S. Pat.** 598147 of Feb. 1, 1898 to Albert Hannemann.

72 [p. 375] **U. S. Pats.** 322802, 322803, 322804 and 322805 of Jul. 21, 1885 to A. G. Day.

73 [p. 375] **Brit. Pats.** 331846 of Apr. 4, 1929 to Frankfurter Gas-Gesellschaft and H. W. Hoelzer; **Ger. Pats.** 200524 of Mar. 26, 1907 to Heinrich Trillich; 425216 of Dec. 28, 1924 to Karl Rösler; 426762 of May 2, 1925 to Karl Rösler; **French Pat.** 780472 of Apr. 26, 1935 to Société du Gaz de Paris.

74 [p. 375] **Ger. Pats.** 119635 of May 6, 1900 to Dr. Zühl & Eisenmann; 119637 of Oct. 9, 1900 to Dr. Zühl & Eisenmann; 122688 of Jul. 29, 1900 to Chem. Fabrik Groszweissandt, G.m.b.H.; 215682 of Sep. 5, 1907 to Ludwig Grote.

75 [p. 375] **U. S. Pats.** 783916 of Feb. 28, 1905 to Georg von Wirkner; 1711448 and 1711449 of Apr. 30, 1929 to O. D. Cunningham; 1847641 of Mar. 1, 1932 to A. B. Cowdery; 1983598 of Dec. 11, 1934 to N. K. Chaney and E. F. Kiefer; **Brit. Pat.** of 1912 (Mar. 11), 6019 to J. Göpper and O. Geiger; **Ger. Pats.** 158731 of Mar. 8, 1903 to C. G. von Wirkner; 247501 of Sep. 7, 1911 to J. Göpper and O. Geiger; 303273 of Feb. 24, 1915 to E. Wirth; 607770 of Oct. 4, 1931 to Ladislao d'Antal; Appl. N–36381 of Mar. 1, 1934 to Hugo Novak; **Swiss Pat.** 181193 of Feb. 17, 1936 to Hugo Novak.

76 [p. 375] **U. S. Pat.** 1968746 of Jul. 31, 1934 to C. O. Condrup; **Can. Pat.** 322527 of May 17, 1932 to Thermal Industrial & Chemical Research Co., Ltd.; **Brit. Pats.** of 1910

(Apr. 1), 7917 to W. E. W. Richards; 319673 and 319718 of Mar. 23, 1928 to László Bolgár; **Ger. Pats.** 543452 of Jan. 26, 1927 to Hermann Meyer; 577520 of Jun. 21, 1930 to Thermal Industrial & Chemical Research Co., Ltd.; 617435 of Aug. 19, 1935 to Ges. für Teerverwertung m.b.H.; 639337 of Oct. 17, 1934 to Ges. für Teerverwertung m.b.H.; 642437 of Jul. 16, 1935 to Ges. für Teerverwertung m.b.H.; **Hungarian Pat.** 107782 of Dec. 15, 1933 to László Bolgár.

77 [p. 375] **Brit. Pat.** 440311 of May 14, 1935 to C. A. Agthe; **Ger. Pats.** Appls. G–88107, of May 16, 1934 to C. A. Agthe; 641393 of May 17, 1934 to C. A. Agthe; 642259 of May 17, 1934 to C. A. Agthe.

78 [p. 375] **Ger. Pat.** 475478 of Feb. 2, 1922 to I. G. Farbenindustrie A.-G.

79 [p. 375] **U. S. Pat.** 599694 of Mar. 1, 1898 to Ferrar Fenton; **Brit. Pat.** of 1896 (Nov. 24), 16548 to Ferrar Fenton; **Ger. Pat.** 288532 of Nov. 29, 1914 to Nicolous Reif.

80 [p. 375] **Brit. Pat.** 391364 of Sep. 23, 1932 to László d'Antal.

81 [p. 375] **Ger. Pat.** 61555 of May 21, 1891 to Georg Friedrich & Co.

82 [p. 375] **Ger. Pats.** 75656 of Dec. 29, 1892 to A. Wendtland; 147163 of Sep. 28, 1902 to Rütgerswerke, A.-G.; 390847 of Aug. 1, 1920 to Walther Schrauth.

83 [p. 376] "Preparation of a New Road Binder," by C. F. Broadhead, *Gas Eng.*, **47**, 349 (1930); *Gas World*, **92**, 621 (1920) also **93**, 298 (1930); *Gas J.*, **190**, 720 (1930); "New Utilizations for Coal Tar," by R. Millet, Industrie Chimique, **22**, 82 (1935); **U. S. Pats.** 1541242 and 1541243 of Jun. 9, 1925 to Homer Behm; 1868879 of Jul. 26, 1932 to C. F. Broadhead and R. S. Andrews; **Brit. Pat.** 334380 of Nov. 29, 1928 to C. F. Broadhead and R. S. Andrews; **Ger. Pats.** 208378 of Sep. 13, 1905 to Otto Schreiber; 439608 of Jun. 6, 1925 to A. Riebeck'sche Montanwerke A.-G.; 526783 of Feb. 20, 1927 to Gesellschaft für Strahlungschemie, G.m.b.H.; **Australian Pat.** 17101 of Nov. 29, 1928 to C. F. Broadhead and R. S. Andrews.

84 [p. 376] **Ger. Pat.** 536552 of Aug. 31, 1926 to Aug. Nowack A.-G.

85 [p. 376] **Brit. Pats.** 438413 of May 14, 1935 to C. A. Agthe; 438415 of May 15, 1935 to C. A. Agthe; 442080 of Jan. 29, 1936 to I. G. Farbenindustrie A.-G.; **Ger. Pats.** 228497 of Nov. 25, 1909 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 406689 of Sep. 24, 1922 to Farbewerke vorm. Meister Lucius & Brüning and G. Balle; 442597 of Oct. 31, 1924 to A. Riebeck'sche Montanwerke A.-G.; **French Pats.** 789948 of Nov. 8, 1935 to C. A. Agthe; 789976 of Nov. 9, 1935 to C. A. Agthe; 794197 of Feb. 10, 1936 to Georges Porot; Addition 46184 to 789948 of Mar. 6, 1936 to C. A. Agthe.

86 [p. 376] **Ger. Pat.** 486475 of Jun. 16, 1925 to Z. M. Stinnes.

87 [p. 376] **U. S. Pat.** 1901872 of Mar. 21, 1933 to William Higburg and P. C. Reilly.

88 [p. 376] **Can. Pat.** 336923 of Nov. 7, 1933 to Barrett Co.

89 [p. 376] **Ger. Pat.** 332888 of Jun. 23, 1924 to Verkaufsvereinigung für Teererzeugnisse, G.m.b.H.

90 [p. 376] **Brit. Pat.** of 1892 (Nov. 19), 21064 to H. Lockwood.

91 [p. 377] **Ger. Pat.** 443075 of Nov. 16, 1924 to E. C. Marcesche.

92 [p. 377] "Naphthol Pitch," by P. M. Grempe, *Färber-Zeitung*, **26**, 322 (1915); *J. Soc. Chem. Ind.*, **35**, 463 (1916); **Ger. Pats.** 302741 and 302742 of Sep. 26, 1916 to Stefan Langguth.

93 [p. 377] **Ger. Pats.** 367364 of Dec. 21, 1921 to Farbwerke vorm. Meister Lucius & Brüning; 378065 of Mar. 23, 1922 to Farbwerke vorm. Meister Lucius & Brüning.

94 [p. 377] **Ger. Pat.** 438743 of Feb. 13, 1925 to K. Scherrer.

95 [p. 377] **Ger. Pat.** 265220 of Jan. 10, 1911 to Gerhard Ley and Ernst Ludwig.

96 [p. 377] "Teer und Pech," by Heinrich Mallison, *Teer*, **22**, 27 (1924).

CHAPTER XVIII

1 [p. 380] "Water-gas Tar Emulsions," by W. W. Odell, Tech. Paper No. 304, Bureau of Mines, Dept. of Interior, Wash., D. C. (1923).

2 [p. 381] "Study of the Composition of Water Gas," by C. R. Downes and A. L. Dean, *J. Ind. Eng. Chem.*, **3**, 108 (1911), also **6**, 366 (1914).

3 [p. 381] "Free Carbon in Water-gas Tars," by J. M. Weiss, *J. Franklin Inst.*, **172**, 277 (1911).

4 [p. 381] **Ger. Pat.** 65239 of Nov. 10, 1891 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich.

5 [p. 383] "Method of Analysis Applicable to Pacific Coast Oil- and Reformed-Gas Tars," by S. C. Schwarz, *Proc. Pacific Coast Gas Assoc.*, **17**, 397 (Aug. 1926).

6 [p. 383] "Blau Gas: A New Gas for Illuminating, Heating and Power Purposes," by William Hallock, *J. Soc. Chem. Ind.*, **27**, 550 (1908); "The Manufacture and Use of Blau Gas," by Hugo Lieber, *Met. Chem. Eng.*, **12**, 153 (1914).

7 [p. 383] **Ger. Pat.** 65239 of Nov. 10, 1891 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jerserich.

8 [p. 383] **Ger. Pat.** 122688 of Jul. 29, 1900 to Chemische Fabrik Gross-Weissandt, G.m.b.H.

CHAPTER XIX

1 [p. 388] "Beitrag zur Fettsäuredestillation, eine Gegenüberstellung der periodischen mit der stetig betriebenen Arbeitsweise," by Otto Krebs, *Teer u. Bitumen*, **28**, 421 (1930).

2 [p. 389] "Heat Requirements for Fatty Acid Distillation," by Victor Mills and R. C. Daniels, *J. Ind. Eng. Chem.*, **26**, 248 (1934).

3 [p. 390] "Stearin Industry," by O. Rosauer, *Chem. Rev. Fett- u. Harz-Ind.*, **15**, 174 (1908).

4 [p. 394] "Cotton-seed Soap Stock," by David Wesson, *J. Soc. Chem. Ind.*, **26**, 595 (1907).

5 [p. 394] **Ger. Pat.** 220582 of Jun. 26, 1909 to Hermann Loeschick.

6 [p. 397] "City Refuse and its Disposal," by H. de B. Parsons, *J. Soc. Chem. Ind.*, **27** 376 (1908); "Collection and Final Disposition of City Wastes by the New York Department of Street Cleaning," by E. D. Very, *J. Soc. Chem. Ind.*, **27**, 378 (1908); "Garbage and Rubbish Disposal in Los Angeles," by S. B. Simons, *Munic. J.*, **38**, 799 (1915).

7 [p. 397] "The Utilization of Sludge from Town Sewage as a Source of Fat," by David Holde, *Seifensieder-Ztg.*, **41**, 1151 (1915).

8 [p. 398] "The Commercial Distillation of Fatty Acids and Wool Grease," by G. F. Pickering, *J. Soc. Chem. Ind.*, **44**, 424T (1925).

9 [p. 399] "Stearin Pitches, Fatty Pitches and Fat Tars," by Carl Stiepel, *Seifensieder-Ztg.*, **62**, 161 and 195 (1935).

10 [p. 400] "Stearin Pitch," by Edward Donath and R. Strasser, *Chem. Z.*, **17**, 1788 (1893); "Die Unterscheidung und chemische Natur von dunkeln pechartigen Rückständen der Destillation von Erdölen, Fetten und Fettsäuren," by David Holde and Julius Marcusson, *Mitt. königl. techn. Versuchanst.* (Berlin), **18**, 147 (1900); *Chem. Rev. Fett- u. Harz-Ind.*, **7**, 2 (1900); "Zur Unterscheidung der Asphalte," by B. M. Margosches, *Chem. Rev. Fett- u. Harz-Ind.*, **11**, 148 (1904); "Examination of Pitches," by Edward Donath and B. M. Margosches, *Chem. Ind.*, **27**, 220 (1904); "Notizen über Stearinpeche," by Edward Donath *Chem. Rev. Fett- u. Harz-Ind.*, **12**, 42 and 73 (1905); "Untersuchung der Kohlenwasserstofföle und Fette," by David Holde, p. 281, Verlag von Julius Springer, Berlin (1913); "Detection of Petroleum Pitch in Fat Distillation Residues," by Julius Marcusson, *Mitt. königl. Materialprüfungsamt*, **30**, 186 (1913); "Stearin Pitch," by H. Mayer, *Seifensieder-Ztg.*, **41**, 394 (1914); "Chemical Technology and Analysis of Oils, Fats and Waxes," by Dr. J. Lewkowitsch, 5th Edition, London (1915); "Stearin Pitch," by Julius Marcusson, *Mitt. königl. Materialprüfungsamt*, **36**, 209 (1918); "Notes on Oil Pitch," by W. Mansbridge, *J. Soc. Chem. Ind.*, **37**, 182T (1918); "Pitches and Bituminous Materials with Special Reference to Stearin Pitches," by H. M. Langton, *Proc. Oil and Color Chem. Assoc.*, **2**, No. 7 (1919); "Relation between the Fat and Asphalt Industries," by Julius Marcusson, *Z. deut. Oel- u. Fett-Ind.*, **41**, 225 1921); "Stearin Pitch," by K. Würth, *Farben-Ztg.*, **30**, 1806 (1925).

11 [p. 401] **Can. Pat.** 113139 of Jul. 21, 1908 to Alfred Vogelgesang; **Brit. Pats.** of 1883 (Jan. 24), 399 to L. A. Groth; of 1906 (Feb. 10), 3345 to J. Connolly; **Ger. Pat.** 217026 of

Apr. 12, 1906 to Alfred Vogelgesang; **French Pat.** 385805 of Dec. 23, 1907 to Marius Dupré and Severin Icard; **Austrian Pat.** 31234 of Dec. 1, 1908 to Kabelfabrik A.-G.

12 [p. 401] **U. S. Pat.** 842615 of Jan. 29, 1907 to Paul Boèrche; **Brit. Pats.** of 1906 (Feb. 10), 3344 and 3345 to J. Connolly.

13 [p. 401] **U. S. Pats.** 529727, 529728, 529729, 529730 and 529905 of Nov. 27, 1894 to William Griscom, Jr.; **Brit. Pats.** of 1894 (Feb. 12), 3045 to L. Bäärnhjelm and A. Jernander; 302710 of Sep. 17, 1927 to J. Y. Johnson; **Ger. Pats.** 77810 of Oct. 20, 1894 to L. Bäärnhjelm and A. Jernander; 225911 of May 25, 1907 to A. F. Malchow; 434143 of Mar. 7, 1923 to Meilach Melamid; 517279 of Dec. 8, 1926 to I. G. Farbenindustrie A.-G.

14 [p. 401] **U. S. Pats.** 1669490 and 1669491 of May 15, 1928 to Wallace Savage.

15 [p. 401] **U. S. Pat.** 1574771 of Mar. 2, 1926 to G. J. Votapek.

16 [p. 401] **U. S. Pat.** 1574842 of Mar. 2, 1926 to C. A. Nash.

17 [p. 401] **Ger. Pat.** 253965 of Aug. 1, 1911 to Isaac Lifschütz.

18 [p. 401] **Ger. Pat.** 277643 of Jun. 6, 1911 to Friedrich Binder.

19 [p. 401] **Can. Pat.** 282877 of Aug. 28, 1928 to I. G. Farbenindustrie A.-G.; **Brit. Pats.** of 1894 (Feb. 12), 3045 to L. Bäärnhjelm and A. Jernander; **Ger. Pats.** 208378 of Sep. 13, 1905 to Otto Schreiber; 414483 of May 24, 1924 to Chem. Laboratorium für Anstrichstoffe, G.m.b.H.

20 [p. 401] **Ger. Pat.** 548348 of Jan. 22, 1929 to S. E. Israel.

21 [p. 401] **U. S. Pat.** 264821 of Sep. 19, 1882 to W. O. Callender; **Ger. Pat.** 81729 of Oct. 14, 1894 to A. Motard & Co.

22 [p. 402] **Brit. Pat.** 374987 of Jun. 23, 1932 to Robert Arnot.

23 [p. 402] **Ger. Pat.** 168048 of Feb. 24, 1904 to Pierre Lacollonge.

24 [p. 403] "The Composition and Examination of Residues Obtained from Fat Distillation," by Julius Marcusson, *Z. angew. Chem.*, **24**, 1297 (1911); "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, p. 63 et seq., Verlag von Wilhelm Engelmann, Leipzig (1921).

25 [p. 403] "Distinguishing between Petroleum Residuums and the Various Fat Pitches," by A. R. Lukens, *Chemist Analyst*, **20**, 3 (1917).

26 [p. 406] "Examination of Pitches," by Edward Donath and B. M. Margosches, *Chem. Ind.*, **27**, 220 (1904).

27 [p. 408] "Glycerin Pitch," by F. M. Grempe, *Seifensieder-Ztg.*, **39**, 1294 (1913); **Ger. Pats.** 309862 of Feb. 11, 1917 to Guldenwerke Chem. Fabrik A.-G.; 312375 of Sep. 29, 1917 to Fr. Steinitzer; 314175 of Jul. 19, 1918 to Wilhelm v. Sperl; 345141 of May 30, 1920 to Chem. Fabrik Plagwitz-Zerbst, G.m.b.H.; 365458 of Sep. 9, 1921 to Meta Sarason.

28 [p. 408] **Ger. Pat.** 528172 of Mar. 26, 1929 to Albert Stahn.

29 [p. 408] **U. S. Pat.** 1424137 of Jul. 25, 1922 to Louis Weisberg and R. S. Potter.

CHAPTER XX

1 [p. 409] "Base of a Crude Oil," by E. C. Lane and E. L. Garton, Report of Investigations R. I. 3279, U. S. Bureau of Mines, Dept. of Interior, Wash., D. C. (Sep., 1935).

2 [p. 411] "Recovering Petroleum from Emulsions by Chemical Treatment," by R. R. Matthews and P. A. Crosby, *J. Soc. Chem. Ind.*, **13** 1015 (1921).

3 [p. 411] **U. S. Pat.** 1373773 of Apr. 5, 1921 to P. T. Sharples.

4 [p. 412] **U. S. Pat.** 1055707 of Mar. 11, 1913 to W. M. Burton.

5 [p. 414] **U. S. Pats.** 781240 of Jan. 31, 1905 to E. A. Starke; 2058913 of Oct. 27, 1936 to Thomas Robinson.

6 [p. 414] "Cut-back Asphalts: Their Characteristics and Use," by Prévost Hubbard, 10th Annual Paving Conference, New Orleans, La. (Dec. 5 to 9, 1932).

7 [p. 417] **Can. Pat.** 309219 of Mar. 10, 1931 to Barrett Co.

8 [p. 418] **U. S. Pats.** 1056980 of Mar. 25, 1913 to R. C. Dundas; 1675462 of Jul. 3, 1928 to A. E. Pew, Jr.; 1842105 of Jan. 19, 1932 to N. E. Loomis.

9 [p. 418] "Applications of Distillation in Modern Petroleum Refining," by J. S. Carey, *J. Und. Eng. Chem.*, **27**, 795 (1935).

10 [p. 426] **U. S. Pats.** 1049667 of Jan. 7, 1913 to W. M. Burton; 1055707 of Mar. 11, 1913 to W. M. Burton; 1105961 of Aug. 4, 1914 to W. M. Burton; 1112113 of Sep. 29, 1914 to W. M. Burton; 1448254 of Mar. 13, 1923 to W. M. Burton.

11 [p. 427] "Fifteen Years of the Burton Process," by R. E. Wilson, *J. Ind. Eng. Chem.*, **20**, 1099 (1928); **U. S. Pat.** 1553847 of Sep. 15, 1925 to W. M. Burton.

12 [p. 428] **U. S. Pat.** 1946949 of Feb. 13, 1934 to C. J. Pratt.

13 [p. 428] **U. S. Pat.** 2045906 of Jun. 30, 1936 to E. W. Gardner, Harry Roden and D. H. Felder.

14 [p. 428] **U. S. Pats.** 1891890 of Dec. 20, 1932 to P. C. Keith, Jr.; 2069620 of Feb. 2, 1937 to H. G. Nevitt.

15 [p. 429] **U. S. Pats.** 2024096 of Dec. 10, 1935 to F. S. Dengler, E. W. Gardner and D. H. Felder; 2068845 of Jan. 26, 1937 to J. O. Collins; **Can. Pat.** 349804 of Apr. 23, 1935 to F. S. Dengler, E. W. Gardner and D. H. Felder; **Brit. Pat.** 456600 of Aug. 3, 1935 to Standard Oil Co. of Cal.

16 [p. 429] **Can. Pat.** 343216 of Jul. 17, 1934 to Standard Oil Development Co.

17 [p. 429] **U. S. Pats.** 1987085 of Jan. 8, 1935 to R. R. Thurston; 2062366 of Dec. 1, 1936 to Charles Mack.

18 [p. 429] **U. S. Pat.** 1993532 of Mar. 5, 1935 to F. J. Stowronski.

19 [p. 429] **U. S. Pat.** 1960885 of May 29, 1934 to R. E. Wilson.

20 [p. 429] "The Cracking Process in the Gas-making Industry," by Gustav Egloff and J. C. Morrell, *J. Ind. Eng. Chem.*, **22**, 1080 (1930).

21 [p. 429] "Tentative Definitions of Terms Relating to Petroleum," (D288—36T) A.S.T.M. Tentative Standards 1936, 669; Approved as American Tentative Standard ASA No:Z 11.28—1936 by the American Standards Assoc.; "Tentative Specifications for Fue Oils," (D 396—34T), A.S.T.M. Tentative Standards 1936, 603.

22 [p. 433] "Characteristics and Differentiation of Native Bitumens and their Residues," by Clifford Richardson, *J. Ind. Eng. Chem.*, **5**, 462 (1913).

23 [p. 433] "Make Asphalt by Vacuum Reduction," by D. H. Bancroft, *Oil & Gas J.*, **29**, 30 (1931); **Can. Pat.** 296045 of Dec. 24, 1929 to Standard Oil Development Co.

24 [p. 434] "Manufacture of Asphalt from Cracking-process Residues," by F. M. H. Taylor, *Trans. Inst. Chem. Eng.*, 3–16 (1931).

25 [p. 434] **U. S. Pat.** 1871207 of Aug. 9, 1932 to W. G. Whitman; **Can. Pat.** 316156 of Oct. 13, 1931 to Standard Oil Co. (Indiana).

26 [p. 434] **U. S. Pat.** 1788792 of Jan. 13, 1931 to R. P. Crothers; **Can. Pat.** 321301 of Apr. 5, 1932 to Universal Oil Products Co.; **Swedish Pat.** 73248 of Dec. 8, 1931 to N. O. Backlund.

27 [p. 434] **U. S. Pats.** 1660294 and 1660295 of Feb. 21, 1928 to R. T. Goodwin; 1812589 of Jun. 30, 1931 to R. T. Goodwin.

28 [p. 434] **U. S. Pat.** 1875823 of Sep. 6, 1932 to C. D. Read; **Can. Pat.** 337017 of Nov. 7, 1933 to Standard Oil Co. (Indiana).

29 [p. 434] **Ger. Pat.** Appl. St–51239 of Aug. 31, 1933 to Standard Oil Development Co.

30 [p. 435] "Asphalt from the Cracking Process," by Gustav Egloff and J. C. Morrell, *J. Ind. Eng. Chem.*, **23**, 679 (1931).

31 [p. 439] **U. S. Pat.** 2025709 of Dec. 31, 1935 to C. M. Baskin; **Can. Pat.** 363897 of Feb. 2, 1937 to Standard Oil Development Co.

32 [p. 439] **U. S. Pat.** 1992752 of Feb. 26, 1935 to W. H. Kershaw.

33 [p. 439] "Quality of Oils for Surface Oiling of Earth Roads and Streets," by F. L. Sperry, Proc. A.S.T.M., **25**, Part II, 376 (1925).

34 [p. 440] "A Practical Treatise on Coal, Petroleum, and Other Distilled Oils," by Abraham Gesner, 2nd Edition, p. 128, Baillière Brothers, New York (1865).

35 [p. 440] *Am. Chemist*, **5**, 359 (1875); **U. S. Pats.** 178061 and 178154 of May 30, 1876 to W. P. Jenney; 190762 of May 15, 1877 to W. P. Jenney.

36 [p. 440] **U. S. Pats.** 101167 of Feb. 22, 1870 to P. N. Mackay; 236995 of Jan. 25, 1881 to E. J. De Smedt.

37 [p. 440] **U. S. Pats.** 237662 of Feb. 8, 1881 to E. J. De Smedt; 239466 of Mar. 29, 1881 to E. J. De Smedt; **Brit. Pat.** of 1881 (Feb. 28), 849 to J. H. Johnson.

38 [p. 440] **U. S. Pat.** 671078 of Apr. 2, 1901 to J. T. Davis.

39 [p. 440] **Ger. Pat.** 32705 of Sep. 25, 1884 to E. Schall.

40 [p. 440] **U. S. Pat.** 298072 of May 6, 1884 to D. H. Dorsett.

41 [p. 440] **U. S. Pat.** 340411 of Apr. 20, 1886 to C. L. Baillard.

42 [p. 440] **U. S. Pat.** 376289 of Jan. 10, 1888 to Heinrich Busse.

43 [p. 440] **U. S. Pats.** 452763 and 452764 of May 19, 1891 to Frederick Salathé; 564341 of Jul. 21, 1896 to Frederick Salathé.

44 [p. 440] **Ger. Pat.** 208378 of Sep. 13, 1905 to Otto Schreiber.

45 [p. 441] "Ueber die Einwirkung von Oxidationsmitteln auf Vaseline und Paraffine," by Alfons Langer, *Chem. Ztg.*, 45, 466 (1921).

46 [p. 441] "The Artificial Production of Asphalt from Petroleum," by C. F. Mabery and J. H. Byerley, *Am. Chem. J.*, 18, 141 (1896); **U. S. Pat.** 524130 of Aug. 7, 1894 to F. X. Byerley; **Can. Pat.** 52049 of Apr. 22, 1896 to F. X. Byerley; **Brit. Pat.** of 1902 (May 14), 11035 to W. Torkington.

47 [p. 441] **U. S. Pat.** 634818 of Oct. 10, 1899 to J. W. Hayward; **Ger. Pat.** 104198 of Sep. 14, 1898 to J. W. Hayward; **French Pat.** 281364 of Sep. 13, 1898 to J. W. Hayward; **Austrian Pat.** 5318 of Oct. 26, 1898 to J. W. Hayward.

48 [p. 441] **U. S. Pats.** 635429 and 635430 of Oct. 24, 1899 to G. F. Culmer and G. C. K. Culmer; 836059 of Nov. 13, 1906 to W. S. Wilkinson; **Can. Pats.** 70574 and 70575 of Mar. 19, 1900 to G. F. Culmer and G. C. K. Culmer; **Ger. Pat.** 125734 of Jun. 9, 1899 to G. F. Culmer and G. C. K. Culmer.

49 [p. 442] "Das Spaltverfahren im theoretischen und praktischen Bilde," by R. Fussteig, *Chem. Ztg.*, 77, (1933).

50 [p. 442] **U. S. Pats.** 2026073 of Dec. 31, 1935 to H. T. Swerissen; **Brit. Pats.** 362580 and 364666 of Oct. 6, 1930 to Burmah Oil Co., Ltd.; **Ger. Pat.** 601019 of Aug. 29, 1931 to Burmah Oil Co., Ltd.; **French Pats.** 719984 of Jul. 11, 1931 to N. V. de Bataafsche Petroleum Maatschappij; 722881 and 722882 of Sep. 14, 1931 to Burmah Oil Co., Ltd.

51 [p. 443] **U. S. Pat.** 1385511 of Jul. 26, 1921 to H. R. Wardell; **Can. Pat.** 215501 of Jan. 31, 1922 to Central Commercial Co.

52 [p. 443] "Blowing Petroleum Residues and Tars," by Julius Marcusson and M. Picard, *Chem. Ztg.*, 48, 338 (1924); **Brit. Pat.** of 1907 (Apr. 2), 7739 to H. F. Catts; **Ger. Pat.** 208378 of Sep. 13, 1905 to Otto Schreiber.

53 [p. 443] **Brit. Pat.** 229690 of Feb. 22, 1924 to K. W. Nielsen; **Ger. Pat.** 437192 of Feb. 21, 1925 to K. W. Nielsen.

54 [p. 443] **U. S. Pat.** 634818 of Oct. 10, 1899 to J. W. Hayward.

55 [p. 443] **U. S. Pat.** 1981384 of Nov. 20, 1934 to Adolf v. Friedolsheim and Martin Luther.

56 [p. 443] **U. S. Pat.** 1997569 of Apr. 16, 1935 to W. A. Craig and F. E. Griffith.

57 [p. 443] "The Oxidation of Mineral Oils by Air," by B. T. Brooks and I. W. Humphrey, *J. Ind. Eng. Chem.*, 9, 746 (1917).

58 [p. 443] **Brit. Pat.** 427777 of Apr. 30, 1935 to Felix Jacobsohn.

59 [p. 443] **Can. Pat.** 319986 of Feb. 23, 1932 to A. Riebeck'sche Montanwerke A.-G.

60 [p. 443] **Brit. Pat.** of 1911 (Nov. 17), 26426 to M. G. C. R. d'Olivier-Mansau; **Ger. Pat.** 625474 of Feb. 10, 1936 to Enterprises Albert Cochery.

61 [p. 443] **U. S. Pat.** 1782186 of Nov. 18, 1930 to Gene Abson.

62 [p. 443] **U. S. Pat.** 1872774 of Aug. 23, 1932 to Martin Luther and Hans Klein.

63 [p. 443] **Japan Pat.** 93247 of Oct. 13, 1931 to Saikiti Mizusima and Teikiti Yamada.

64 [p. 444] **U. S. Pat.** 1513133 of Oct. 28, 1924 to A. J. Rowland.

65 [p. 444] **U. S. Pats.** 1995324 of Mar. 26, 1935 to W. B. D. Penniman; 2029504 of Feb. 4, 1936 to E. G. Ragatz; **Brit. Pat.** 364666 of Oct. 6, 1930 to Burmah Oil Co., Ltd.

66 [p. 444] **U. S. Pats.** 646639 of Apr. 3, 1900 to J. A. Dubbs; 1057227 of Mar. 25, 1913 to J. A. Dubbs; **Can. Pat.** 324794 of Aug. 2, 1932 to N. V. de Bataafsche Petroleum Maat-

schappij; **Brit. Pat.** 363972 of Jul. 10, 1931 to N. V. de Bataafsche Petroleum Maatschappij; **Austrian Pat.** 118641 of Mar. 5, 1930 to Karl Bauer and Artur Urman.

67 [p. 447] "Behavior of High-boiling Mineral Oils on Heating in Current of Air and Steam," by Fritz Schreiber, *Z. angew. Chem.*, **23**, 99 (1910).

68 [p. 444] **U. S. Pats.** 81071 of Aug. 18, 1868 to F. L. de Gerbeth, 1430538 of Oct. 3, 1922 to H. H. Culmer.

69 [p. 444] **Brit. Pat.** 334032 of Oct. 23, 1928 to N. V. de Bataafsche Petroleum Maatschappij; **Ger. Pat.** 437192 of Feb. 21, 1925 to K. W. Nielsen.

70 [p. 444] **Can. Pat.** 297208 of Feb. 4, 1930 to E. W. Hultman.

71 [p. 444] "Effect of Blowing Petroleum Residues with Mixtures of Air and Chlorine," by Walter Rosenbaum, Thesis Univ. of Cincinnati (Ohio), (May, 1932). **U. S. Pats.** 1979676 and 1979677 of Nov. 6, 1934 to O. R. Douthett.

72 [p. 444] "The Chlorination of Natural Asphalts," by H. Loebell, *Chem. Rev. Fett- u Harz-Ind.*, **18**, 165 (1911).

73 [p. 444] **Brit. Pats.** 370367 of Oct. 2, 1929 to Georgij Tesch; 456600 of Nov. 12, 1936 to Standard Oil Co. of Cal.; **French Pat.** 793501 of Jan. 27, 1936 to Standard Oil Co. of Cal.

74 [p. 444] "Continuous Oxidation of Asphaltic Residues," by A. D. Demchenko, *Neftyanoe Khozyaĭstvo*, **17**, 704 (1929).

75 [p. 444] **U. S. Pats.** 1448070 of Mar. 13, 1923 to H. R. Wardell; 1950900 of Mar. 13, 1934 to E. B. McConnell; 1999018 of Apr. 23, 1935 to E. W. Gard and B. G. Aldridge; **Can. Pat.** 355374 of Jan. 14, 1936 to E. B. McConnell.

76 [p. 445] **U. S. Pats.** 1404435 of Jan. 24, 1922 to Emile Gevers-Orban; 1718679 of Jun. 25, 1929 to J. C. Black and W. D. Rial.

77 [p. 445] **U. S. Pat.** 1865081 of Jun. 28, 1932 to M. L. Chappell and T. H. Dowlen.

78 [p. 445] **U. S. Pat.** 1886380 of Nov. 8, 1932 to T. H. Dowlen.

79 [p. 445] **U. S. Pats.** 1902305 of Mar. 21, 1933 to Lester Kirschbraun; 1912667 of Jun. 6, 1933 to H. T. Swerissen; 1942656 of Jan. 9, 1934 to C. P. McNeil; 1953333 of Apr. 3, 1934 to J. C. Black and W. D. Rial; 1953345 and 1953346 of Apr. 3, 1934 to E. W. Gard and B. G. Aldridge; 1973294 of Sep. 11, 1934 to Erik Mutter; 2032546 of Mar. 3, 1936 to C. P. McNeil and S. A. Montgomery; 2057265 of Oct. 13, 1936 to H. I. Ringgenberg; **Can. Pats.** 324340 of Jul. 19, 1932 to Flintkote Corp.; 339532 of Feb. 20, 1934 to Shell Development Co.; **Brit. Pat.** 407522 of Feb. 25, 1933 to Burmah Oil Co., Ltd.

80 [p. 445] **U. S. Pats.** 1586376 of May 25, 1926 to J. S. Miller, Jr.; 1715069 of May 28, 1929 to Lester Kirschbraun; 1830925 of Nov. 10, 1931 to O. C. Brewster and W. M. Perry; 1889697 of Nov. 29, 1932 to H. B. Pullar; 1909335 of May 16, 1933 to O. C. Brewster and W. M. Perry; 1975563 of Oct. 2, 1934 to Sijbren Tijmstra; 1982920 of Dec. 4, 1934 to E. B. McConnell; 1988766 of Jan. 22, 1935 to B. G. Aldridge; 2002670 of May 28, 1935 to E. B. McConnell; 2055459 of Sep. 22, 1936 to Adolf Gutzwiller; **Can. Pat.** 324340 of Jul. 19, 1932 to Flintkote Corp.; **Brit. Pats,** 163363 and 163656 of Nov. 18, 1919 to V. C. Illing and J. Kelly; 337130 of Sep. 12, 1929 to J. Y. Johnson; 361158 of Dec. 17, 1930 to Standard Oil Co.; **French Pat.** 791278 of Dec. 6, 1935 to N. V. de Bataafsche Petroleum Maatschappij.

81 [p. 445] **U. S. Pats.** 1673533 of Jun. 12, 1928 to E. A. Rudigier; 1766446 of Jun. 24, 1930 to M. R. L. Miller; **Can. Pat.** 348888 of Mar. 12, 1935 to Standard Oil Co.; **Ger. Pat.** 285388 of Dec. 4, 1928 to Standard Oil Development Co.

82 [p. 445] **U. S. Pat.** 1766446 of Jun. 24, 1930 to M. R. L. Miller; **Can. Pat.** 304904 of Oct. 21, 1930 to M. R. L. Miller.

83 [p. 445] **U. S. Pat.** 1714206 of May 21, 1929 to J. C. Black and W. D. Rial.

84 [p. 445] **U. S. Pat.** 1911114 of May 23, 1933 to E. W. Gard and Philip Subkow; **Brit. Pat.** 398112 of Mar. 11, 1933 to Soc. pour l'Expolit des Proc. Ab-der-Halden.

85 [p. 445] **U. S. Pat.** 1774756 of Sep. 2, 1930 to A. F. MacLachlan; **Brit. Pat.** 334032 of Jul. 30, 1929 to N. V. de Bataafsche Petroleum Maatschappij; **Dutch Pat.** 25260 of Oct. 15, 1931 to N. V. de Bataafsche Petroleum Maatschappij.

86 [p. 445] **U. S. Pat.** 2046081 of Jun. 30, 1936 to C. P. McNeil.

87 [p. 445] **U. S. Pats.** 998569 of Jul. 18, 1911 to C. B. Forward; 1409237 of Mar. 14, 1922

to H. B. Pullar; **Brit. Pat.** of 1911 (Apr. 20), 9646 to C. B. Forward; **Ger. Pats.** 208378 of Sep. 13, 1905 to Otto Schreiber; 290792 of Feb. 28, 1914 to C. B. Forward.

88 [p. 445] **U. S. Pat.** 1709242 of Apr. 16, 1929 to H. A. Winkelmann.

89 [p. 445] **Brit. Pats.** 338538 of Aug. 20, 1929 to László d'Antal; 362034 of Sep. 15, 1930 to J. Y. Johnson.

90 [p. 445] **U. S. Pat.** 2028922 of Jan. 28, 1936 to B. L. Rose.

91 [p. 445] **U. S. Pat.** 1306520 of Jun. 10, 1919 to C. J. Burkley; **Ger. Pat.** 431782 of Jul. 8, 1924 to Ges. für Gas-und Teerverwertung, m.b.H.; **French Pat.** 799625 of Jun. 16, 1936 to N. V. de Bataafsche Petroleum Maatschappij.

92 [p. 445] **U. S. Pat.** 1889365 of Nov. 29, 1932 to Albert Loebel; **Dutch Pat.** 28709 of Jan. 16, 1933 to N. V. de Bataafsche Petroleum Maatschappij.

93 [p. 445] **U. S. Pats.** 1937749 of Dec. 5, 1933 to A. R. Ebberts; 2067264 of Jan. 12, 1937 to A. R. Ebberts.

94 [p. 445] **Ger. Pat.** 336603 of May 26, 1918 to Harburger Chemische Werke.

95 [p. 445] **U. S. Pat.** 1417837 of May 30, 1922 to Lester Kirschbraun.

96 [p. 446] "Value of Blown Asphalts and Their Manufacture," by H. B. Pullar, *Good Roads*, 3, 146 (1912).

97 [p. 446] "Oxidized Bitumen," by J. Manheimer, *Petroleum*, 31, No. 40, 4 (1935).

98 [p. 448] **U. S. Pats.** 1948926 of Feb. 20, 1934 to R. E. Haylett; 2073088 of Mar. 9, 1937 to A. P. Anderson and W. K. Nelson; **French Pat.** 804313 of Oct. 21, 1936 to Socony-Vacuum Oil Co.

99 [p. 448] **U. S. Pats.** 1990466 of Feb. 12, 1935 to H. L. Allan; 2076799 of Apr. 13, 1937 to R. R. Thurston.

100 [p. 448] **U. S. Pat.** 2026039 of Dec. 31, 1935 to G. R. Hoover.

101 [p. 448] **U. S. Pat.** 1881753 of Oct. 11, 1932 to Albert Loebel.

102 [p. 451] "Ueber geblasene Erdölpeche," by David Holde and S. Weill, *Petroleum Z.*, 19, 451 (1923); "The Reactions Involved in Blowing Petroleum Residual Oils and Tars," by Julius Marcusson and M. Picard, *Chem. Ztg.*, 48, 338 (1924).

103 [p. 452] "Concerning Blown Petroleum Asphalts," by Edmund Graefe, *Petroleum*, 14 (May 10, 1923).

104 [p. 452] **U. S. Pat.** 1513133 of Oct. 28, 1924 to A. J. Rowland.

105 [p. 452] **U. S. Pats.** 58615 of Oct. 9, 1866 to A. G. Day; 165530 of Jul. 13, 1875 to H. R. Bellamy; Reissues 6707 and 6708 of Oct. 19, 1875 to A. G. Day; 210405, 210406, 210407, 210408 and 210409 of Dec. 3, 1878 to A. G. Day; 322802, 322803, 322804 and 322805 of Jul. 21, 1885 to A. G. Day.

106 [p. 452] **U. S. Pats.** 468867 of Feb. 16, 1892 to J. A. Dubbs; 480234 and 480235 of Aug. 2, 1892 to J. A. Dubbs; 608372 of Aug. 2, 1898 to J. A. Dubbs; **Can. Pats.** 38929 of May 12, 1892 to J. A. Dubbs; 41025 of Nov. 26, 1892 to J. A. Dubbs; 41101 of Nov. 29, 1892 to J. A. Dubbs.

107 [p. 452] **U. S. Pats.** 452760 of May 19, 1891 to Frederick Salathé; 580139 of Apr. 6, 1897 to William Morison; 667786 of Feb. 12, 1901 to F. A. Lobert; 896558 of Aug. 18, 1908 to F. A. Lobert.

108 [p. 452] **U. S. Pat.** 1163593 of Dec. 7, 1915 to C. N. Forrest; **Can. Pat.** 171345 of Aug. 15, 1916 to Barber Asphalt Paving Co.; **Brit. Pat.** of 1897 (May 24), 12829 to H. C. P. Graves; **Ger. Pat.** 294046 of Dec. 3, 1915 to Barber Asphalt Paving Co.; **French Pat.** 480271 of Jul. 12, 1916 to Barber Asphalt Paving Co.

109 [p. 453] **U. S. Pat.** 264821 of Sep. 19, 1882 to W. O. Callender; **Brit. Pat.** of 1881 (Oct. 11), 4408 to W. O. Callender.

110 [p. 453] **U. S. Pats.** 90084 of May 18, 1869 to J. E. Dotch and Edward Duempelmann; 411171 of Sep. 17, 1889 to C. A. A. H. Siebert; 540792 of Jun. 11, 1895 to J. A. Just; 624081 and 624082 of May 2, 1899 to D. W. Peck; 626479 of Jun. 6, 1899 to P. C. Bell; 1020643 of Mar. 19, 1912 to Carleton Ellis; 1264932 of May 7, 1918 to G. A. Henderson; 1409088 of Mar. 7, 1922 to R. G. Erwin; 1505206 of Aug. 19, 1924 to Hugh Knight; 1830486 of Nov. 3, 1931 to S. S. Sadtler; **Can. Pats.** 338120 of Dec. 26, 1933 to John Mosley; 348823 of Mar. 12,

1935 to Canadian Industries, Ltd.; **Brit. Pat.** 188354 of Jul. 4, 1921 to Bitumul Matita S. A. Romana; **Ger. Pats.** 215682 of Sep. 5, 1907 to Ludwig Grote; 385283 of Jan. 23, 1921 to André Manéa.

111 [p. 453] **U. S. Pats.** 376289 of Jan. 10, 1888 to Heinrich Busse; 411171 of Sep. 17, 1889 to C. A. A. H. Siebert; 425412 of Apr. 15, 1890 to I. T. Dyer; 626479 of Jun. 6, 1899 to P. C. Bell; 714858 and 714859 of Dec. 2, 1902 to W. R. Brixey; 728851 of May 26, 1903 to W. R. Brixey; 1888270 of Nov. 20, 1932 to J. S. Hipple; **French Pat.** 779332 of Apr. 2, 1935 to O. A. Hubert and Pierre Lambert.

112 [p. 453] **U. S. Pats.** 529727, 529728, 529729 and 529730 of Nov. 27, 1894 to William Griscom; **Brit. Pat.** of 1894 (Nov. 27), 22966 to W. P. Thompson.

113 [p. 453] **U. S. Pat.** 1846107 of Feb. 23, 1932 to D. McK. Hepburn, S. S. Sadtler and E. F. Cayo.

114 [p. 453] **U. S. Pat.** 651358 of Jun. 12, 1900 to J. A. Just; **Ger. Pat.** 433854 of Oct. 20, 1925 to K. Sautermeister and F. Wilhelm.

115 [p. 453] **U. S. Pat.** 716787 of Dec. 23, 1902 to R. M. Thompson.

116 [p. 453] **U. S. Pat.** 210405 of Dec. 3, 1878 to D. T. Day.

117 [p. 453] **U. S. Pat.** 1674287 of Jun. 19, 1928 to Frank Kurath and L. T. Richardson.

118 [p. 453] **U. S. Pats.** 3598 of May 25, 1844 to Edouard Deutsch; 309450 of Dec. 16, 1884 to M. A. Fenner; 540791 and 540792 of Jun. 11, 1895 to J. A. Just; 624081 and 624082 of May 2, 1899 to D. W. Peck; 724882 of Apr. 7, 1903 to Victor de Karavodine; 1326110 of Dec. 23, 1919 to E. L. Sharpneck; **Can. Pats.** 49751 and 49752 of Aug. 24, 1895 to J. A. Just; **Brit. Pat.** of 1898 (Nov. 22), 24630 to A. E. Alexander; **Ger. Pats.** 109499 of Dec. 11, 1898 to M. Kaempff; 124629 of Apr. 29, 1899 to D. W. Peck; 145250 of Sep. 25, 1901 to Victor de Karavodine; 253377 of Jul. 19, 1911 to Albin Baer.

119 [p. 453] **Can. Pat.** 337586 of Dec. 5, 1933 to Sri Krishna.

120 [p. 453] **U. S. Pat.** 1417837 of May 30, 1922 to Lester Kirschbraun.

121 [p. 453] **U. S. Pat.** 90825 of Jun. 1, 1869 to J. E. Dotch and Eduard Duempelmann; **Ger. Pat.** 33101 of Mar. 3, 1885 to Ernst Doerr.

122 [p. 453] **Ger. Pat.** 596366 of Jul. 11, 1935 to Anton Breuer; **French Pat.** 764460 of May 22, 1934 to Anton Breuer.

123 [p. 453] **U. S. Pat.** 499354 of Jun. 13, 1893 to J. de S. Brown.

124 [p. 453] **Ger. Pat.** 83550 of Jun. 6, 1894 to Albert Hannemann and G. Boisly.

125 [p. 453] **Ger. Pat.** 517166 of Aug. 11, 1926 to Andrei Manéa.

126 [p. 453] **Ger. Pat.** 625210 of Aug. 16, 1932 to László d'Antal.

127 [p. 453] **U. S. Pat.** 276891 of May 1, 1883 to J. G. Sanderson; **Brit. Pats.** 256513 of Mar. 15, 1926 to E. C. R. Marks; 331846 of Apr. 4, 1929 to Frankfurter Gas-Gesellschaft and H. W. Hoelzer; 397864 of Feb. 22, 1932 to William Griffiths and Frank Foulger; **Ger. Pat.** 200524 of Mar. 26, 1907 to Heinrich Trillich.

128 [p. 453] **U. S. Pats.** 322802, 322803, 322804 and 322805 of Jul. 21, 1885 to D. T. Day; 616047 of Dec. 13, 1898 to C. E. Anthony.

129 [p. 453] **U. S. Pats.** 1464480 of Aug. 7, 1923 to G. A. Henderson and R. G. Erwin; 1464481 and 1464482 of Aug. 7, 1923 to G. A. Henderson.

130 [p. 453] **U. S. Pats.** 90825 of Jun. 1, 1869 to J. E. Dotch and Edward Duempelmann; 1678635 of Jul. 31, 1928 to O. A. Cherry and C. F. Chosa; **Ger. Pats.** 119635 of May 6, 1900 to Dr. Zühl & Eisemann; 119637 of Oct. 9, 1900 to Dr. Zühl & Eisemann; 410012 of Mar. 15, 1923 to Farbwerke vorm. Meister Lucius & Brüning. **French Pat.** 781118 of May 9, 1935 to O. A. Hubert and Pierre Lambert.

131 [p. 453] **Ger. Pat.** Appl. S–109094 of Apr. 20, 1933 to Bauunternehmung Sager & Woerner Strassenbau, G.m.b.H.

132 [p. 453] **Ger. Pat.** 228497 of Nov. 25, 1909 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jerserich.

133 [p. 453] **Brit. Pat.** 397864 of Aug. 22, 1933 to William Griffiths and Frank Foulger.

134 [p. 453] **U. S. Pat.** 1003040 of Sep. 12, 1911 to F. H. Dunham.

135 [p. 453] **Can. Pat.** 147095 of Apr. 8, 1913 to W. M. Burton; **Ger. Pat.** Appl. B-147.775 of Jan. 13, 1931 to C. H. Borrmann.

136 [p. 453] **U. S. Pat.** 688073 of Dec. 3, 1901 to A. W. Dow.

137 [p. 454] **U. S. Pats.** 1194750 of Aug. 15, 1916 to Lester Kirschbraun; 1436450 of Nov. 21, 1922 to Lester Kirschbraun; 1735503 of Nov. 12, 1929 to Lester Kirschbraun.

138 [p. 454] **U. S. Pat.** 284098 of Aug. 28, 1883 to R. S. Waring.

139 [p. 454] **U. S. Pat.** 239260 of Mar. 22, 1881 to J. I. Livingston.

140 [p. 454] **U. S. Pat.** 1599369 of Sep. 7, 1926 to S. W. Moss.

141 [p. 454] **U. S. Pat.** 611620 of Oct. 4, 1898 to C. B. Forward and J. M. Davidson.

142 [p. 454] **Ger. Pat.** 582074 of Jan. 3, 1931 to N. V. de Bataafsche Petroleum Maatschappij.

143 [p. 455] "Some Relationships between the Characteristics of Steam-distilled Petroleum Asphalts." by B. A. Anderton, Proc. A.S.T.M., **20**, Part II, 298 (1920).

144 [p. 456] "Characteristics and Differentiation of Native Bitumens and their Residuals," by Clifford Richardson, *Eng. Record*, **67**, 466 (1913).

145 [p. 457] "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, pp. 99 and 255, Verlag von Wilhelm Engelmann, Leipzig (1921).

146 [p. 457] "Distinction of Natural Asphalt Bitumen from Petroleum Pitch and Coal-tar Pitch," by Jenö Kovács and S. Söbét, *Chem. Rev. Fett- u. Harz-Ind.*, **7**, 8 (1900); "Investigations on Asphalt," by Georg Lunge and V. Křepelka, *Chem. Ztg.*, **28**, 177 (1904); "Detection of Adulterants in Natural Asphaltum," by Basilius Malenkovic, *Oester. Chem. Zeit.*, **8**, 123 (1905); "Distinction between Natural and Petroleum Asphalts," by Julius Marcusson and R. Eichmann, *Chem. Zeit.*, **32**, 965 (1908); "Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 276, John Wiley & Sons, New York (1908); "Identifying Asphalts," by Julius Marcusson, *Chem. Rev. Fett- u. Harz-Ind.*, **18**, 47 (1911); "Composition and Examination of Fat Distillation Residues," by Julius Marcusson, *Z. angew. Chem.*, **24**, 1297 (1911); "Petroleum Asphalts," by D. Lohmann, *Chej. Rev. Fett- u. Harz-Ind.*, **18**, 107 (1911); "Chemical Composition and Methods of Distinguishing Natural and Artificial Asphalts," by Julius Marcusson, *Chem. Rev. Fett- u. Harz-Ind.*, **19**, 166 (1912); "Separation of Natural and Petroleum Asphalts," by Julius Marcusson, *Chem. Zeit.*, **36**, 801 (1912); "Quantitative Determination of Natural Asphaltum in the Presence of Artificial Asphaltum," by Julius Marcusson, *Z. angew. Chem.*, **26**, 91 (1913); "Detection of Natural Asphalt and Petroleum Pitch," by F. Schwarz, *Chem. Rev. Fett- u. Harz-Ind.*, **20**, 28 (1913); "Analysis of Petroleum Oil and Mineral Wax," by H. Kantorowicz, *Chem. Zeit.*, **37**, 1394, 1438, 1565 and 1594 (1913); "Differentiation of Natural and Artificial Asphalts," by Julius Marcusson, *Mitt. k. Materialprüf*, **32**, 419 (1914); "Differentiation of Natural and Oil Asphalts," by E. C. Pailler, *J. Ind. Eng. Chem.*, **6**, 286 (1914); "Chemistry and Analysis of Asphaltum," by Julius Marcusson, *Chem. Zeit.*, **38**, 813 and 822 (1914); "Untersuchungen über natürliche und künstliche Asphalte," by Julius Marcusson, *Kunstoffe*, **5**, 75–78, 90–92, 101–102 (1915); *Petroleum Z.*, **10**, 373 (1915); "Zur Unterscheidung von Natur- und Kunst-asphalte," by Edmund Graefe, *Z. angew. Chem.*, **29**, 21 (1916); "Chemical Composition of Natural Asphalts," by Julius Marcusson, *Z. angew. Chem.*, **29**, 346 and 349 (1916); "Chemical Composition of Artificial Asphalts," by Julius Marcusson, *Z. angew. Chem.*, **31**, 113 and 119 (1918); "Notes on Oil Pitch," by W. Mansbridge, *J. Soc. Chem. Ind.*, **37**, 182T (1918); "Bitumen Substitutes or Artificial Asphalts, Especially Mineral Oil Pitch and Other Pitches," by G. Buchner, *Seifensieder Ztg.*, **46**, 385 and 407 (1919); "Distinction between Natural Asphalts and Oil Asphalts," by E. C. Pailler, *Caoutchouc and Gutta Percha*, **16**, 10063 (1919); "Ueber geblasene Erdölpeche," by David Holde and S. Weill, *Petroleum Z.*, **19**, 451 (1923).

147 [p. 458] "The California Asphaltum Industry," by F. H. Minard, *Eng. Mining J.*, **75**, 503 (1903); "Production and Use of Petroleum in California," by L. E. Aubury, Bull. No. 32, California State Mining Bureau, San Francisco, Cal. (Mar., 1904).

148 [p. 458] "The Properties of Bitumens Poor and Rich in Paraffine," by J. Manheimer, *Petroleum*, **28**, No. 16, 6 (1932).

149 [p. 461] **Can. Pat.** 309385 of Mar. 17, 1931 to László Bolgár; **Swiss Pat.** 146276 of Jan. 28, 1929 to László d'Antal.

150 [p. 461] **U. S. Pat.** 2016721 of Oct. 8, 1935 to P. W. Kunze.

151 [p. 461] **U. S. Pat.** 2016721 of Oct. 8, 1935 to P. W. Kunze; **Brit. Pat.** 359461 of Jul. 21, 1930 to R. E. Ellis.

152 [p. 461] **Can. Pat.** 341991 of May 29, 1934 to Shell Development Co.

153 [p. 461] "Utilization of Asphaltic Base Acid-Sludge Obtained in Refining Petroleum and Shale Oils," by Charles Baskerville, *J. Ind. Eng. Chem.*, **12**, 30 (1920); **U. S. Pat.** 1231985 of Jul. 3, 1917 to Charles Baskerville.

154 [p. 461] **U. S. Pat.** 1862060 of Jun. 7, 1932 to F. R. Moser and Jacques Greutert.

155 [p. 461] "Manufacture of Asphalt from Acid Sludge," by F. W. Padgett, J. W. Donnell and R. I. Jacobi, *Petroleum Engr.*, **4**, 18 (1933).

156 [p. 461] **U. S. Pat.** 1271387 of Jul. 2, 1918 to R. de M. Taveau.

157 [p. 461] **Brit. Pat.** of 1913 (Apr. 5), 8069 to Ludwig Landsberg.

158 [p. 462] "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, p. 49, Verlag von Wilhelm Engelmann, Leipzig (1921).

159 [p. 464] **U. S. Pat.** 1547252 of Jul. 28, 1925 to P. W. Kunze.

160 [p. 464] **Ger. Pat.** 585766 of Apr. 24, 1931 to László d'Antal.

161 [p. 464] **U. S. Pat.** 1917869 of Jul. 11, 1933 to J. C. Bird.

162 [p. 464] **Brit. Pat.** 319210 of Sep. 11, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

163 [p. 465] **U. S. Pat.** 1745155 of Jan. 28, 1930 to J. F. Faber and M. L. Chappell.

CHAPTER XXI

1 [p. 468] **U. S. Pats.** 117946 of Aug. 8, 1871 to A. B. Vandemark; Reissue 4591 of Oct. 10, 1871 to A. B. Vandemark; 198260 of Dec. 18, 1877 to N. B. Abbott; 214312 of Apr. 15, 1879 to A. T. Perry; 227080 of Apr. 27, 1880 to C. M. Warren; 234442 of Nov. 16, 1880 to C. M. Warren; 356411 of Jan. 18, 1887 to Terrence McSweeney.

2 [p. 469] **U. S. Pat.** 1417838 of May 30, 1922 to Lester Kirschbraun.

3 [p. 469] **U. S. Pat.** 339787 of Apr. 13, 1886 to E. D. Kendall.

4 [p. 470] "Synthetic Resins from Petroleum Hydrocarbons," by C. A. Thomas, and W. H. Carmody, *J. Ind. Eng. Chem.*, **24**, 1125 (1932); **U. S. Pats.** 1836629 of Dec. 15, 1931 to C. A. Thomas and C. A. Hochwalt; 1852043 of Apr. 5, 1932 to Max Deseniss and Andreas Nielsen.

5 [p. 470] **U. S. Pat.** 1868211 of Jul. 19, 1932 to J. W. Le Nobel; **Brit. Pat.** 313433 of Aug. 30, 1928 to N. V. de Bataafsche Petroleum Maatschappij; **Ger. Pats.** 555970 of Sep. 18, 1928 to N. V. de Bataafsche Petroleum Maatschappij; Appl. St–50015 of Oct. 26, 1932 to Standard Oil Development Co.; **Dutch Pat.** 24396 of Jul. 15, 1931 to J. W. Le Nobel.

6 [p. 471] **U. S. Pat.** 1926523 of Sep. 12, 1933 to S. C. Fulton and Vladimir Kalichevsky.

7 [p. 471] **Brit. Pat.** 339470 of Nov. 25, 1929 to D. Anderson & Son, Ltd. and R. O. Child.

8 [p. 471] "Synthetic Resins from Petroleum Hydrocarbons," by C. A. Thomas and W. H. Carmody, *J. Ind. Eng. Chem.*, **24**, 1125 (1932); "Resins from Cracked Distillates," by C. A. Thomas and F. J. Soday, *J. Ind. Eng. Chem.*, **28**, 1174 (1936); **U. S. Pats.** 1836629 of Dec. 15, 1931 to C. A. Thomas and C. A. Hochwalt; 1939932 of Dec. 19, 1933 to C. A. Thomas; 1947626 of Feb. 20, 1934 to C. A. Thomas; 1981824 of Nov. 20, 1934 to S. C. Fulton; 1982707 and 1982708 of Dec. 4, 1934 to C. A. Thomas; 2023495 of Dec. 10, 1935 to C. A. Thomas; 2029288 of Feb. 4, 1936 to U. B. Bray; 2039363, 2039365 and 2039367 of May 5, 1936 to C. A. Thomas; 2055486 of Sep. 29, 1936 to S. C. Fulton; **Can. Pat.** 333230 of Jun. 13, 1933 to C. A. Thomas; **Brit. Pat.** 340001 of Mar. 12, 1931 to C. A. Thomas.

9 [p. 471] **U. S. Pats.** 2052172 of Aug. 25, 1936 to P. K. Frolich; 2052173 of Aug. 25, 1936 to P. K. Frolich, Carl Winning and S. C. Fulton.

CHAPTER XXII

1 [p. 472] **U. S. Pats.** 616047 of Dec. 13, 1898 to C. E. Anthony; 617706 of Jan. 17, 1899 to C. E. Anthony; 620082 of Feb. 21, 1899 to C. E. Anthony; 655130 and 655131 of Jul. 31, 1900 to R. M. Thompson; 716787 of Dec. 23, 1902 to R. M. Thompson; 734482 and 734483 of Jul. 21, 1903 to S. R. Whitall; 768101 of Aug. 23, 1904 to F. M. Whitall; 864836 of Sep. 3, 1907 to W. F. Doerflinger and L. H. Buck; 903275 of Nov. 10, 1908 to W. F. Doerflinger and

L. H. Buck; 984240 of Feb. 14, 1911 to J. C. Ross; 1057093 and 1057094 of Mar. 25, 1913 to J. C. Ross; 1259260 of Mar. 12, 1918 to Archibald Maxwell; 1332359 of Mar. 2, 1926 to R. R. Rosenbaum; 1653766 of Dec. 27, 1927 to R. E. Haughey.

2 [p. 472] **U. S. Pats.** 292770 of Jan. 29, 1884 to P. H. Vander Weyde; 903275 of Nov. 10, 1908 to W. F. Doerflinger and L. H. Buck; **Brit. Pat.** of 1907 (Sep. 2), 19629 to M. Stine.

3 [p. 475] "Recent Uses of Elaterite," by Elihu Palmer, *Eng. Mining J.*, **102**, 856 (1916).

4 [p. 475] **U. S. Pat.** 292770 of Jan. 29, 1884 to P. H. Vander Weyde.

CHAPTER XXIII

1 [p. 481] "Factors Controlling the Behavior of Asphalt Mixtures," by J. C. Morrell and Lester Kirschbraun, *Chem. Met. Engr.*, **29**, 362 (1923).

2 [p. 483] **Ger. Pat.** 325780 of Jul. 7, 1918 to Chem.-analyt.-Lab., Schlicht & Horák.

3 [p. 483] **Brit. Pat.** 433826 of Jul. 12, 1934 to Alexander Wacker Ges. für Elektrochemische Industrie, G.m.b.H.

4 [p. 483] **Ger. Pats.** 623041 of Jul. 24, 1934 to Chemische Fabrik Flörsheim Dr. H. Noerdlinger A.-G.; 624043 of Nov. 23, 1934 to Chemische Fabrik Flörsheim Dr. H. Noerdlinger A.-G.

5 [p. 483] **Ger. Pat.** 394107 of Nov. 25, 1921 to Sudfeldt & Co.

6 [p. 483] **Brit. Pat.** 429548 of May 31, 1934 to H. D. Elkington.

7 [p. 483] **Can. Pat.** 361536 of Nov. 3, 1936 to I. G. Farbenindustrie A.-G.; **Brit. Pat.** 445473 of Oct. 9, 1934 to I. G. Farbenindustrie A.-G.; **Ger. Pats.** 623400 of Dec. 21, 1935 to I. G. Farbenindustrie A.-G.; 638920 of Sep. 29, 1935 to I. G. Farbenindustrie A.-G.; 639607 of Dec. 22, 1935 to I. G. Farbenindustrie A.-G.; Appl. I-53297 of Sep. 28, 1935 to I. G. Farbenindustrie A.-G.

8 [p. 483] **Brit. Pat.** 421113 of Apr. 6, 1933 to Eugéne Roualt.

9 [p. 483] **French Pat.** 788834 of Oct. 18, 1935 to J. L. Chambon and G. M. Hart.

10 [p. 483] "Introfiers, or Impregnation Accelerators," by Marc Darrin, *J. Ind. Eng. Chem.*, **20**, 801 (1928); **Brit. Pat.** 312909 of Jun. 2, 1928 to N. V. de Bataafsche Petroleum Maatschappij.

11 [p. 483] **Brit. Pat.** 365511 of Aug. 13, 1930 to C. G. Fox.

12 [p. 483] **Ger. Pats.** 572640 of Aug. 17, 1929 to Amber Size & Chemical Co., Ltd.; 573696 of Apr. 5, 1933 to Amber Size & Chemical Co., Ltd.

13 [p. 483] **U. S. Pat.** 1849867 of Mar. 15, 1932 to C. R. Eckert.

14 [p. 484] "Beiträge zur Teerstrassenbauforschung, insonderheit Studien über die Mischung von Teer und Asphalt," by Hans Lüer, Allgemeiner Industrie-Verlag, G.m.b.H., Berlin (1928); "Properties of Tar-Petroleum-Asphalt Mixtures," by P. Schläpfer, *Petroleum*, **25**, 1457 and 1585 (1929); **U. S. Pats.** 21553 of Sep. 21, 1858 to G. W. Cushing; 281999 of Jul. 24, 1883 to J. B. Hyde; 290057 of Dec. 11, 1883 to J. B. Hyde; **Can. Pats.** 29959 of Oct. 8, 1888 to Thomas Bryce; 40860 of Nov. 2, 1892 to C. T. Williams; 42045 of Feb. 18, 1893 to C. T. Williams; 63056 of May 15, 1899 to Hector Bernier; **Ger. Pat.** Appl. Sch-100973 of Apr. 15, 1933 to Theodor Schmidt.

15 [p. 484] "A Further Study of the Heterogeneity of Asphalt—A Quantitative Method," by G. L. Oliensis, *Proc.* A.S.T.M., **36**, Part II, (1936).

16 [p. 484] **Brit. Pat.** of 1913 (Mar. 18), 1458 to J. J. Ingham; **Ger. Pats.** 552732 of Apr. 19, 1928 to Frankfurter Gasgesellschaft and H. W. Hoelzer; 562486 of Feb. 24, 1929 to Frankfurter Gasgesellschaft and H. W. Hoelzer.

17 [p. 484] "Asphalt, Bitumen and Tar," by A. W. Attwooll and D. C. Broome, *Roads and Road Construction*, Sep. and Oct. (1931).

18 [p. 484] "Non-homogeneous Mixtures of Tars and Petroleum Asphalts," by Franz Macht, *Asphalt u. Teer*, **31**, 325, 352, 416 and 435 (1931); *Bitumen*, **73**, (1931); "Die Teer-Erdölbitumen-Mischungen für den Strassenbau," by G. H. Klinkmann, Thesis, Karlsruhe (1931).

19 [p. 484] **U. S. Pat.** 228960 of Jun. 15, 1880 to C. M. Warren.

20 [p. 484] "The Importance of Anthracene Oil as a Constituent of Bituminous Road-Building Material," by Albrecht von Skopnik, *Teer u. Bitumen*, **29**, 527 (1931).

21 [p. 484] **Brit. Pats.** 355937 of Aug. 18, 1930 to Amber Size & Chemical Co., Ltd.; 356003 of Oct. 13, 1930 to Amber Size & Chemical Co., Ltd.

22 [p. 484] **Ger. Pat.** 342421 of Jan. 29, 1920 to Diethelm Beck; 557375 of Mar. 26, 1931 to Georg Wieland; **French Pats.** 797717 and 797718 of May 2, 1936 to "Straba" Strassenbaudearfs A.-G.

23 [p. 484] **U. S. Pats.** 472352 of Apr. 5, 1892 to I. Hill; 1233415 and 1233416 of Jul. 17, 1917 to W. H. Steinberg; 1847805 of Mar. 1, 1932 to Robert Arnot; **Brit. Pat.** 389280 of Mar. 30, 1932 to C. F. Broadhead and R. S. Andrews.

24 [p. 484] **Can. Pat.** 328338 of Dec. 6, 1932 to Texaco Development Corp.

25 [p. 485] "Ueber den Zusatz von Asphaltbitumen zu Steinkohlenteeren," by H. Walther, *Teer u. Bitumen*, **29**, 165 (1931); "Untersuchung des Einflusses von Bitumenzusatz zu Teer an Probekörpern," by Ing. Knipping, *Teer u. Bitumen*, **33**, 151 (1935).

26 [p. 485] **U. S. Pats.** 1327572 of Jan. 6, 1920 to H. D. Ryan; 1643520 and 1643521 of Sep. 27, 1927 to C. S. Reeve; 1649545 of Nov. 15, 1927 to G. F. Renou; Reissue 17762 of Aug. 5, 1930 to C. S. Reeve; **Can. Pats.** 223205 of Aug. 29, 1922 to H. D. Ryan; 284743 of Nov. 13, 1928 to Australian Bituminous Compounds, Ltd.; **Ger. Pat.** 412423 of Nov. 25, 1919 to Shale Reduction Machinery Corporation.

27 [p. 485] **U. S. Pats.** 1875458 of Sep. 6, 1932 to W. H. Hill; 1875502 of Sep. 6, 1932 to H. J. Rose and W. H. Hill; 1905060 of Apr. 25, 1933 to H. J. Rose and W. H. Hill; **Can. Pat** 318878 of Jan. 12, 1932 to Koppers Co.; **Brit. Pats.** 131588 of Aug. 8, 1919 to Rütgerswerke, A.-G.; 268372 of Mar. 25, 1927 to Koppers Co.; 356239 of May 31, 1930 to Koppers Co.; 358988 of Apr. 11, 1930 to Koppers Co.; 362934 of Sep. 5, 1930 to Koppers Co.; **Ger. Pat.** 579033 of Jun. 20, 1933 to Koppers Co.

28 [p. 485] **U. S. Pat.** 1916333 of Jul. 4, 1933 to H. J. Rose; **Can. Pat.** 321900 of Apr. 26, 1932 to Koppers Co.; **Brit. Pat.** 353495 of Apr. 17, 1932 to Koppers Co.; **Ger. Pat.** 551548 of May 13, 1930 to Koppers Co.

29 [p. 485] **U. S. Pat.** 1993343 of Mar. 5, 1935 to E. V. Harlow; **Can. Pat.** 326955 of Oct. 18, 1932 to Koppers Co.; **Brit. Pat.** 353493 of Apr. 17, 1932 to Koppers Co.

30 [p. 485] **Ger. Pat.** 110302 of Jul. 11, 1899 to Vereinigte Electricitäts A.-G.

31 [p. 485] **Ger. Pat.** Appl. L–77372 of Jan. 27, 1931 to Paul Lechler.

32 [p. 486] "Valuation of Natural and Artificial Bitumens for Insulating Cables," by F. Dupré, *Chem. Ztg.*, **42**, 445 (1918); *J. Soc. Chem. Ind.*, **37**, 645A (1918).

33 [p. 486] **Ger. Pats.** 508960 of Oct. 3, 1926 to László Bolgár; Appl. L–77372 of Jan. 27, 1931 to Paul Lechler.

34 [p. 486] **Ger. Pat.** 367364 of Dec. 21, 1921 to Farbwerke vorm. Meister Lucius & Brüning.

35 [p. 486] **U. S. Pat.** 1332359 of Mar. 2, 1920 to R. R. Rosenbaum.

36 [p. 486] "Incorporation of Rubber Derivatives with Tar Products," Part II, by D. D. Pratt and R. Handley, *Bull. Rubber Growers' Assoc.*, **18**, 391 (1936); **Brit. Pat.** 434559 of Sep. 27, 1934 to J. R. Geigy A.-G.; **Ger. Pat.** 621970 of Sep. 28, 1933 to J. R. Geigy A.-G.

37 [p. 487] "Asphalt-Rubber Mixtures," by F. C. van Heurn and M. A. Begheyn, *Kolloid Z.*, **66**, 219 (1934); "Incorporation of Rubber with Tar Products," by D. D. Pratt and R. Handley, *Bull. Rubber Growers' Assoc.*, **17**, 245 (1935); "The Physical Properties of Rubber-Bitumen Mixtures," by W. S. Davey, *J. Soc. Chem. Ind.*, **55**, 43T (1936); "An Asphalt-Rubber Compound, by Yukio Uno and Masao Ishida, *J. Soc. Chem. Ind.* Japan, **39**, 90B (1936); "Incorporation of Rubber Derivatives with Tar Products," by D. D. Pratt and Roy Handley, *Bull. Rubber Growers' Assoc.*, **18**, 391 (1936); **Brit. Pat.** 434317 of Dec. 12, 1934 to International Vereeniging voor de Rubber-en Andere Cultures in Nederlandsch-Indië.

38 [p. 487] **U. S. Pat.** 2052607 of Sep. 1, 1936 to A. B. Cowdery.

39 [p. 487] **U. S. Pat.** 1849867 of Mar. 15, 1932 to C. R. Eckert; 2054243 of Sep. 15, 1936 to A. B. Cowdery; **Can. Pats.** 362541, 362542 and 362543 of Dec. 15, 1936 to Barrett Co.

40 [p. 487] **U. S. Pat.** 1911131 of May 23, 1933 to F. C. Langenberg, H. S. Hunt and H. G. Reddick.

41 [p. 487] **U. S. Pat.** 1769629 of Jul. 1, 1929 to A. C. Fischer.

42 [p. 488] **Can. Pat.** 312536 of Jun. 23, 1931 to Kaspar Winkler.

43 [p. 488] **Ger. Pat.** 605234 of Nov. 7, 1934 to Chemieprodukte, G.m.b.H.

44 [p. 488] **U. S. Pat.** 1997261 of Apr. 9, 1935 to C. D. Lowry, Jr.

45 [p. 489] "Physical Characteristics of a Paint Coating," by R. S. Perry, Am. Inst. of Architects, Michigan Chapter (Jun. 4, 1907).

46 [p. 489] **Ger. Pat.** 566939 of Mar. 29, 1928 to Richard Grün.

47 [p. 489] **U. S. Pat.** 102824 of May 10, 1870 to H. W. Johns.

48 [p. 489] **Ger. Pat.** 574527 of Apr. 13, 1933 to Alexander Supan.

49 [p. 489] **Brit. Pat.** 355430 of Jun. 30, 1930 to N. V. de Bataafsche Petroleum Maatschappij and H. D. Elkington; **French Pats.** 718507 of Jun. 11, 1931 to N. V. de Bataafsche Petroleum Maatschappij; 772201 of Oct. 25, 1934 to N. V. de Bataafsche Petroleum Maatschappij.

50 [p. 492] "Effect of Overheating Asphalts," by C. J. Frankforter, *J. Ind. Eng. Chem.*, **2**, 239 (1910); "Some Relations of the Effect of Overheating to Certain Physical and Chemical Properties of Asphalts," by A. W. Hixson and H. E. Hands, *J. Ind. Eng. Chem.*, **9**, 651 (1917); "The Effect of the Mixing Temperature on the Physical Properties of an Oil Asphalt in a Sheet Asphalt Mixture," by J. H. Bateman and H. L. Lehmann, Proc. A.S.T.M., **29**, II, 943 (1929); "Veränderung der physikalischen Eigenschaften des Bitumens bei Herstellung von Walzasphaltgemischen mit verschiedenen Temperaturen," by Ernst Gerlach, *Teer u. Bitumen*, **34**, 381 (1936).

CHAPTER XXIV

1 [p. 494] **U. S. Pat.** 1783955 of Dec. 9, 1930 to M. T. Chamberlain; **Ger. Pat.** 579958 of Nov. 7, 1930 to Société de Recherches et de Perfectionnements Industriels.

2 [p. 494] **U. S. Pats.** 1448370 of Mar. 13, 1923 to H. R. Wardell; 1464480 of Aug. 7, 1923 to G. A. Henderson and R. G. Erwin; 1834950 of Dec. 8, 1931 to Fred Jaeger; 1854100 of Apr. 12, 1932 to A. D. Brito; **Can. Pat.** 307000 of Dec. 23, 1930 to Patent & Licensing Corp.; **Brit. Pat.** 394122 of May 5, 1932 to "Straba" Strassenbaubedarfs A.-G.; **Ger. Pat.** 604340 of Jun. 1, 1932 to "Straba" Strassenbaubedarfs A.-G.; 642320 of Mar. 29, 1933 to "Straba" Strassenbaubedarfs A.-G.; Appl. Sch-93848 of Apr. 15, 1931 to Friedrich Schmidt; Appl. St-48984 of Feb. 24, 1932 to "Straba" Strassenbaubedarfs A.-G.

3 [p. 494] **Ger. Pats.** 332941 of Jan. 3, 1920 to Nicolaus Reif; 363810 of Nov. 1, 1921 to Nicolaus Reif.

4 [p. 495] **Ger. Pats.** 117758 of Jun. 27, 1899 to Bernhard Löhr; 139610 of Nov. 19, 1901 to Schliemann & Co.

5 [p. 495] **U. S. Pats.** 1603502 of Oct. 19, 1926 to D. B. W. Alexander; 1857802 of Sep. 6, 1932 to J. W. Clark; 1894630 of Jan. 17, 1933 to H. C. Neuberger.

6 [p. 496] **U. S. Pat.** 1176006 of Mar. 21, 1916 to Reinhold Wallbaum.

7 [p. 496] **Can. Pat.** 324499 of Jul. 26, 1932 to Barrett Co.

8 [p. 496] **U. S. Pats.** 63873 of Apr. 16, 1867 to Alburtis Eagle; 1546335 of Jul. 14, 1925 to F. A. Browne; 1986871 of Jan. 8, 1935 to J. A. Withee.

9 [p. 496] **Brit. Pat.** 216911 of Feb. 5, 1923 to T. H. Butler.

10 [p. 496] **U. S. Pats.** 34404 of Feb. 18, 1862 to Joseph Clarke and David French; 51309 of Dec. 5, 1865 to Louis Groneweg, J. H. Pulte and C. T. Jones; 57626 of Aug. 28, 1866 to Joseph Darby; 57999 of Sep. 11, 1866 to J. P. Thompson; 58148 of Sep. 18, 1866 to Jesse Stow and James White; 58169 of Sep. 18, 1866 to J. G. Holliday; 60309 of Dec. 4, 1866 to J. J. Wiggin; 62619 of Mar. 5, 1867 to G. L. Eagan and C. H. Eagan; 62936 of Mar. 19, 1867 to John Calflisch; 63367 of Apr. 2, 1867 to Joseph Darby; 71731 of Dec. 3, 1867 to R. H. Fitts; 74083 of Feb. 4, 1868 to David Harger; 79732 of Jul. 7, 1868 to F. A. Curtis; 83539 of Oct. 27, 1868 to Michael Pinner; 83576 of Oct. 27, 1868 to J. J. Wiggin; 91223 of Jun. 15, 1869 to John Fullager and Miles Byrne; 93482 of Aug. 10, 1869 to S. I. Russell and J. H. Cole; 94495 of

Sep. 7, 1869 to G. E. Hopkins; 103403 of May 24, 1870 to L. A. Wilder; 114577 of May 9, 1871 to Thomas Madeley; 169385 of Nov. 2, 1875 to L. P. Teed; 652065 of Jun. 19, 1900 to Karl Wessel; 770137 of Sep. 13, 1904 to H. F. Williams; 785650 of Mar. 21, 1905 to J. H. Amies; 934495 of Sep. 21, 1909 to J. H. Amies; 935573 of Sep. 28, 1909 to J. H. Amies; 951471 of Mar. 8, 1910 to J. H. Amies; 966982 of Aug. 9, 1910 to J. H. Amies; 1150481 of Aug. 17, 1915 to J. H. Amies; 1234919 of Jul. 31, 1917 to P. J. McNamara; 1409088 of Mar. 7, 1922 to R. G. Erwin; 1456492 of May 22, 1923 to D. M. Hepburn; 1985718 of Dec. 25, 1934 to J. F. Carle; **Can. Pats.** 37609 of Oct. 15, 1891 to J. H. Farr; 166227 of Nov. 23, 1915 to P. J. McNamara; **Brit. Pat.** 395288 of Jul. 13, 1933 to Maschinenfabrik "Komnick," G.m.b.H.; **Ger. Pat.** 120785 of Jun. 22, 1899 to H. O. Köhler; 619973 of Oct. 10, 1935 to Maschinenfabrik "Komnick," G.m.b.H.; Appl. A-61495 of Apr. 8, 1931 to A. T. Arnot; **Australian Pat.** 25040 of Feb. 7, 1930 to B. H. Stone.

11 [p. 496] **Ger. Pat.** 96094 of Jan. 28, 1898 to Richard Joecks and Johannes Joecks.

12 [p. 496] **U. S. Pat.** 1771017 of Jul. 22, 1930 to Shannon Smith.

13 [p. 496] **U. S. Pat.** 1771795 of Jul. 29, 1930 to T. F. King; 1991196 of Feb. 12, 1935 to A. C. Deer; **Ger. Pat.** 596584 of Aug. 1, 1929 to T. F. King.

14 [p. 496] **Can. Pats.** 279581 of Oct. 21, 1914 to Wilhelm Reppin; 289610 of Jan. 8, 1916 to Wilhelm Reppin.

15 [p. 496] **U. S. Pat.** 1320672 of Nov. 4, 1919 to J. E. Black and M. A. Popkess.

16 [p. 496] **U. S. Pat.** 1418905 of Jun. 6, 1922 to K. L. Binkley; **Fr. Pat.** 320198 of Apr. 4, 1902 to Fritz Basenau.

17 [p. 496] **U. S. Pat.** 1887518 of Nov. 15, 1932 to S. S. Sadtler.

18 [p. 496] **U. S. Pat.** 1847805 of Mar. 1, 1932 to Robert Arnot.

19 [p. 496] **U. S. Pat.** 1603502 of Oct. 19, 1926, to D. B. W. Alexander.

20 [p. 496] **U. S. Pats.** 1198769 and 1198955 of Sept. 19, 1916 to Clifford Richardson; 1240252 and 1240253 of Sep. 18, 1917 to M. A. Popkess; 1320709 of Nov. 4, 1919 to M. A. Popkess; 1327726 of Jan. 13, 1920 to S. R. Murray; **Can. Pat.** 164824 of Sep. 14, 1915 to Clifford Richardson; **Brit. Pat.** 58396 of Jun. 20, 1935 to Wales Dove Bitumastic, Ltd.

21 [p. 496] **U. S. Pat.** 1237852 of Aug. 21, 1917 to C. L. V. Zimmer; **Can. Pat.** 155273 of Apr. 21, 1914 to Reinhold Wallbaum; **Swiss Pat.** 71890 of Mar. 1, 1916 to C. L. V. Zimmer.

22 [p. 496] **U. S. Pats.** 164233 of Jun. 8, 1875 to Eli Thayer; **Ger. Pats.** 111088 of Mar. 2, 1899 to Adolf Gentszch; 312085 of Jul. 15, 1916 to C. L. V. Zimmer; 336603 of May 26, 1918 to Harburger Chemische Werke, Schön & Co., and W. Daitz.

23 [p. 496] "Valuation of Natural and Artificial Bitumens for Insulating Cables," by F. Dupré; *Chem. Ztg.*, **42**, 445 (1918); **Ger. Pat.** 330650 of Jul. 6, 1918 to Harburger Chemische Werke, Schön & Co., and W. Daitz.

24 [p. 496] **French Pat.** 735631 of Apr. 21, 1932 to Cyprien Laffitte.

25 [p. 496] **Brit. Pat.** 404939 of Jul. 18, 1932 to A. B. Manning.

26 [p. 496] "Gelatinized Tars and Bitumens," by Da Fano, Asfalti, **6**, 40 (1934); **Brit. Pat.** 404966 of Jan. 19, 1933 to Erich Koch.

27 [p. 497] **Brit. Pats.** 312372 of Nov. 18, 1927 to J. J. V. Armstrong; 316897 of Mar. 3, 1928 to South Metropolitan Gas Co.

28 [p. 497] **U. S. Pats.** 60830 of Jan. 1, 1867 to J. F. Boynton; 1158418 of Oct. 26, 1915 to T. M. Hart; 1190615 of Jul. 11, 1916 to H. P. Willis; 1206076 of Nov. 28, 1916 to Oscar Altpeter; 1252433 of Jan. 8, 1918 to T. M. Hart; 1315342 of Sep. 9, 1919 to E. L. Sharpneck; 1328310 of Jan. 20, 1920 to H. P. Willis; 1372467 of Mar. 22, 1921 to H. P. Willis; 1455598 of May 15, 1923 to W. S. Barrie and Lawrence Chadwick; 1505880 of Aug. 19, 1924 to A. M. Fairlie; 1558082 of Oct. 20, 1925 to Daniel Gardner; 1677272 of Jul. 17, 1928 to F. L. Carson; 1769208 of July 1, 1930 to T. C. Crowley; **Can. Pats.** 11864 of Oct. 9, 1880 to Harvey Hall; 11976 of Nov. 13, 1880 to James Butler; 13798 of Dec. 4, 1881 to James Butler; 162145 of Apr. 20, 1915 to H. P. Willis; 217416 of Apr. 4, 1922 to W. S. Barrie and Lawrence Chadwick; **Brit. Pat.** 213659 of Jan. 2, 1923 to S. Burton.

29 [p. 497] "Some Obseirvations on the Colloidal Character of Asphalts," by R. E. Kirk and L. H. Ryerson, *J. Phys. Chem.*, **29**, 865 (1925); **U. S. Pats.** 1464480 of Aug. 7, 1923 to G. A. Henderson and R. G. Erwin; 1464481 of Aug. 7, 1923 to G. A. Henderson.

30 [p. 497] "The Formation of Asphalt in Petroleum," by R. Zaloziecki and C. Zielinski, 8th Intern. Cong. Applied Chem., 10, 335 (1912); "The Action of Anhydrous Stannic Chloride on Petroleum and its Distillates," by Gregory Torossian, *J. Ind. Eng. Chem.*, 13, 903 (1921); "Action of Zinc Chloride and Sulphuric Acid on Coal Tar," by F. K. Chéou and F. Yung, Contr. Inst. Chem. Nat. Acad. Peiping, 1, 153 (1934); "Special Coal Tar for Road-paving," by F. K. Chéou and H. L. Chang, *Bull. Nat. Acad. Peiping*, 5, No. 1, 23 (1934); "Asphaltic Plastic Masses," by S. Shapiro, Novosti Tekhniki, Seiya Gornorudnaya Prom., 7 6 (1935); **U. S. Pats.** 811563 of Feb. 6, 1906 to J. P. Ihart; 1379053 of May 24, 1921 to E. L. Sharpneck; 1545440 of Jul. 7, 1925 to Heinrich Neumann; 1720487 of Jul. 9, 1929 to Richard Lichtenstern; 1720929 of Jul. 16, 1929 to Hermann Staudinger and Herman Bruson; 1769795 of Jul. 1, 1930 to W. G. Leamon; 2045741 of Jun. 30, 1936 to Carl Winning and R. M. Thomas; **Can. Pat.** 14768 of May 12, 1882 to Antonio Pelletier; **Brit. Pats.** of 1906 (May 17), 11523 to E. W. Edmunds, A. A. Goodell and William Jones; of 1913 (Feb. 26), 4896 to J. Göpper and O. Geiger; 270274 of Apr. 13, 1927 to N. V. de Bataafsche Petroleum Maatschappij; 320579 of Jan. 16, 1929 to Richard Lichtenstern; 325816 of Nov. 27, 1928 to Emil Rahman; 330565 of Mar. 9, 1929 to N. V. Mijnbouw-en Cultuur-Maatschappij "Boeton"; 382842 of Feb. 27, 1932 to Paul Lechler; 442080 of Jul. 28, 1934 to I. G. Farbenindustrie A.-G.; **Ger. Pats.** 312064 of Jun. 21, 1918 to Eduard Herzinger and Edmund Naylor; 358626 of Jun. 25, 1920 to A. Heuffel; 578934 of Jan. 15, 1929 to Richard Lichtenstern; Appl. L–79860 of Nov. 23, 1931 to Paul Lechler; **Fr. Pats.** 536172 of Mar. 29, 1921 to Heinrich Neumann; 670796 of Mar. 5, 1929 to N. V. Mijnbouw-en Cultuur-Maatschappij "Boeton"; 688542 of Apr. 11, 1929 to Cie. des Mines de Vicoigne, Noeux et Drocourt; 730570 of Jan. 27, 1932 to Paul Lechler; **Austrian Pats.** 72401 of Sep. 11, 1916 to Göpper and O. Geiger; 117051 of Nov. 15, 1929 to Erwin Bergel.

31 [p. 497] "The Formation of Asphalt in Petroleum," by R. Zaloziecki and C. Zielinski, 8th Intern. Cong. Applied Chem., 10, 335 (1912); "The Catalytic Oxidation of Petroleum Oils," by C. E. Waters, *J. Ind. Eng. Chem.*, 13, 901 (1921); **U. S. Pats.** 403631 of May 21, 1889 to A. T. Woodward; 407271 of Jul. 16, 1889 to A. T. Woodward; 503425 of Aug. 15, 1893 to J. W. Kidwell; 538614 of Apr. 30, 1895 to J. W. Kidwell; 1020801 of May 19, 1912 to T. E. Conway; 1505206 of Aug. 19, 1924 to Hugh Knight; 1505880 of Aug. 19, 1924 to A. M. Fairlie; **Can. Pat.** 297208 of Feb. 4, 1930 to E. W. Hultman; **Brit. Pats.** of 1914 (Jan. 23) 1805 to E. A. White; 307465 and 307466 of Mar. 8, 1929 to Kaspar Winkler; **Austrian Pat.** 122973 of Jan. 15, 1931 to Gustav Müller and Johannes Fischer.

32 [p. 497] **U. S. Pats.** 1379052 of May 24, 1921 to E. L. Sharpneck; 1394874 of Oct. 25, 1921 to G. A. Wilson, Jr.; **Brit. Pat.** of 1893 (Sep. 18), 17514 to William Smith; **Ger. Pat.** 503922 of Jan. 30, 1927 to Combustion Utilities Corp.; **Fr. Pat.** 327959 of May 13, 1902 to Emile Jasset.

33 [p. 497] **U. S. Pats.** 733797 of Jul. 14, 1903 to Fritz Basenau and Ali Cohen; 1634241 of Jun. 28, 1927 to C. N. Forrest; **Brit. Pat.** of 1902 (Apr. 7), 8071 to Fritz Basenau; **Ger. Pat.** 139845 of Mar. 20, 1902 to Fritz Basenau; **Fr. Pat.** 320198 of Apr. 4, 1902 to M. Basenau; **Austrian Pat.** 41781 of Dec. 1, 1909 to E. S. Ali Cohen.

34 [p. 497] **U. S. Pat.** 433215 of Jul. 29, 1890 to Israel Rabinowicz.

35 [p. 497] **U. S. Pat.** 1720487 of Jul. 9, 1929 to Richard Lichtenstern; **Brit. Pat.** 320579 of Jan. 16, 1929 to Richard Lichtenstern.

36 [p. 497] **Brit. Pat.** 340293 of Jul. 27, 1929 to J. Y. Johnson.

37 [p. 497] **U. S. Pats.** 610626 of Sep. 13, 1898 to P. H. Hansen; 1238528 of Aug. 28, 1917 to Ernst Krause; 1641637 of Sep. 6, 1927 to L. C. Lutyens and R. O. Child; **Brit. Pats.** 238586 of Apr. 11, 1924 to L. C. Lutyens; 268950 of Apr. 14, 1927 to R. O. Child; 291858 of Aug. 16, 1927 to D. Anderson & Son, Ltd. and R. O. Child; **Ger. Pats.** 38221 of Feb. 17, 1886 to Philipp Cornely; 240482 of May 1, 1910 to A.-G. für Asphaltierung und Dachbedeckung, vorm. Johannes Jeserich; 437238 of Apr. 10, 1925 to L. C. Lutyens and R. O. Child.

38 [p. 497] **U. S. Pat.** 2057042 of Oct. 13, 1936 to W. W. McLaurin; **Brit. Pat.** 323060 of Oct. 6, 1928 to F. N. Nicholls.

39 [p. 497] **Brit. Pat.** 378051 of Feb. 6, 1931 to T. F. King.

40 [p. 497] **Ger. Pat.** 277653 of Apr. 17, 1910 to Julius Stockhausen.

41 [p. 497] **Ger. Pats.** 580356 of Jul. 10, 1933 to Hanseatische Mühlenwerke, A.-G.; 605234 of Nov. 7, 1934 to Chemieprodukte, G.m.b.H.; Appl. H-125634 of Feb. 21, 1931 to Hanseatische Mühlenwerke, A.-G.; **French Pat.** 705505 of Nov. 5, 1930 to S. W. Aretz.

42 [p. 497] **U. S. Pat.** 505531 of Sep. 26, 1893 to E. M. McCook; **Ger. Pat.** 332629 of Feb. 5, 1919 to A. Hambloch.

43 [p. 497] "Diatomaceous Earth," by Robert Calvert, Chemical Catalog Co., Inc., New York (1930); "Diatomite," by Paul Hatmaker, Information Circular I. C.-6391, U. S. Bureau of Mines, Dept. of Commerce, Wash., D. C. (Feb. 1931); "Kieselguhr, ihre Gewinnung, Veredlung und Anwendung," by Franz Krczil, Verlag von Ferdinand Enke, Stuttgart (1936); **U. S. Pats.** 227352 of May 11, 1880 to Edgar Everhart; 416791 of Dec. 10, 1889 to J. I. Livingston and William Griscom; 437033 of Sep. 23, 1890 to R. S. Merrill; 446654 of Feb. 17, 1891 to H. F. Williams; 1446178 of Feb. 20, 1923 to T. F. Harrington; 1519560 of Dec. 16, 1924 to Charles Swan; 1632456 of Jun. 14, 1927 to Charles Swan; 2042964 of Jun. 2, 1936 to H. H. Rinehart; **Can. Pat.** 289219 of Apr. 30, 1929 to Charles Swan; **Brit. Pats.** 372940 of May 5, 1921 to A. B. C. Dahlberg; 434221 of Aug. 28, 1935 to Pipe Linings, Ltd.; 443471 of May 23, 1935 to Berry, Wiggins & Co., Ltd.; **Ger. Pats.** 3097 of Mar. 7, 1878 to F. A. Malchow; 125891 of Nov. 24, 1900 to Hans Christen; 443230 of Jan. 15, 1925 to J. U. A. Ohlsen.

44 [p. 497] **U. S. Pats.** 99277 of Feb. 1, 1870 to T. R. Abbott; 225679 of Mar. 16, 1880 to A. T. Woodward; 298072 of May 6, 1884 to D. H. Dorsett; 403631 of May 21, 1889 to A. T. Woodward; 407271 of Jul. 16, 1889 to A. T. Woodward; 420648 of Feb. 4, 1890 to J. B. Williams; 1452309 of Apr. 17, 1923 to Gustav Monrath; 2009146 of Jul. 23, 1935 to G. A. New; **Can. Pat.** 5298 of Oct. 30, 1875 of J. B. Slichter; **Ger. Pat.** 64680 of Sep. 23, 1891 to Bruno Roedelius.

45 [p. 497] **Can. Pat.** 321794 of Apr. 26, 1932 to H. J. Phillips.

46 [p. 497] **U. S. Pats.** 56363 of Jul. 17, 1866 to F. M. Buell; 58975 of Oct. 23, 1866 to Franklin Bearse and G. E. Hopkins; 59551 of Nov. 13, 1866 to W. H. H. Burnham; 70478 of Nov. 5, 1867 to G. O. Smith and J. H. Smith; 73645 of Jan. 21, 1868 to S. B. Pierce and Pembroke Pierce; 77177 of Apr. 28, 1868 to Charles de Hass; 85453 of Dec. 29, 1868 to John Kivett and George Kivett; 86355 of Feb. 2, 1869 to J. W. Brown; 107756 of Sep. 27, 1870 to Charles Burgess; 113628 of Apr. 11, 1871 to John Cipperley; 124117 of Feb. 27, 1872 to D. G. Conger; 126026 of Apr. 23, 1872 to D. G. Conger; 142595 of Sep. 9, 1873 to Albert Thiele; 159629 of Feb. 9, 1875 to A. N. Atwood; 181528 of Aug. 29, 1876 to C. L. Fowler; 298072 of May 6, 1884 to D. H. Dorsett; 352445 of Nov. 9, 1886 to J. W. Butler; 415864 of Nov. 26, 1889 to S. H. Gilson; 692627 of Feb. 4, 1902 to B. B. Clawson; 77173 of Dec. 13, 1904 to E. I. Allison; **Can. Pats.** 1808 of Nov. 21, 1872 to Elias Burnham; 2725 of Sep. 11, 1873 to Elias Burnham; 2726 of Sep. 12, 1873 to Elias Burnham; **Brit. Pats.** of 1871 (Apr. 19), 1035 to W. E. Newton; of 1898 (Nov. 14), 23981 to J. H. W. Stringfellow; of 1903 (Oct. 15), 22216 to A. E. Tanner and E. A. Claremont; of 1910 (Apr. 23), 9933 to R. C. Sharp; 36935 of Feb. 21, 1931 to F. W. Valle-Jones and C. J. W. Cloke; 252802 of Mar. 7, 1925 to J. A. W. Pine.

47 [p. 497] **Brit. Pat.** of 1902 (Nov. 12), 24864 to W. W. Pilkington and W. R. Ormandy; **Ger. Pat.** 18902 of Sep. 13, 1881 to A. T. Woodward.

48 [p. 498] **U. S. Pats.** 51309 of Dec. 5, 1865 to Louis Groneweg, J. H. Pulte and C. T. Jones; 57324 of Aug. 21, 1866 to Benjamin Hinkley; 59551 of Nov. 13, 1866 to W. H. H. Burnham; 60830 of Jan. 1, 1867 to J. F. Boynton; 76012 of Mar. 24, 1868 to E. B. Wells; **Ger. Pats.** 113896 of Jul. 12, 1899 to B. Reichelt and H. Schultze; 238842 of Dec. 24, 1909 to S. J. Sauvageot; **Swedish Pat.** 80490 of May 23, 1934 to K. E. Karlsson.

49 [p. 498] **U. S. Pats.** 62810 of Mar. 12, 1867 to A. F. Boon; 819833 of May 8, 1906 to E. J. Yetter; 820162 and 820163 of May 8, 1906 to E. J. Yetter; **Ger. Pat.** 335775 of Aug. 14 1919 to A. Kauffmann.

50 [p. 498] **U. S. Pat.** 1188850 of Sep. 12, 1871 to C. L. Fowler.

51 [p. 498] "Improvement of the Quality of Plastic Masses on an Asphaltic Base," by G. V. Kagan, Ukrain, *Chem. J.*, 11, 165 (1936); **Can. Pats.** 314787 of Sep. 1, 1931 to Aluminiumerz Bergbau und Industrie, A.-G.; 325435 of Aug. 30, 1932 to W. A. Dyes; **Brit. Pats.** 356797 of Jun. 10, 1930 to Aluminiumerz Bergbau und Industrie, A.-G.; 357293 of Jul. 2, 1930 to Aluminiumerz Bergbau und Industrie, A.-G.; **Ger. Pats.** 568112 of Jun. 11, 1929 to

Aluminiumerz Bergbau und Industrie, A.-G.; 570350 of Mar. 22, 1930 to Aluminiumerz Bergbau und Industrie, A.-G. **Fr. Pat.** 693867 of Apr. 14, 1930 to Aluminiumerz Bergbau und Industrie, A.-G.

52 [p. 498] "Georgia and Alabama Clays as Fillers," by W. M. Weigel, Tech. Paper No. 343, U. S. Dept. of Commerce, Bureau of Mines, Wash., D. C. (1925); **U. S. Pats.** 38833 of Jun. 9, 1863 to Isaac Marsh; 42925 of May 31, 1864 to Stephen Bowerman; 48311 of Jun. 20, 1865 to Alfred Robinson; 51309 of Dec. 5, 1865 to Louis Groneweg, J. H. Pulte and C. T. Jones; 56444 of Jul. 17, 1866 to W. L. Potter; 57400 of Aug. 21, 1866 to C. Spear; 57999 of Sep. 11, 1866 to J. P. Thompson; 58169 of Sep. 18, 1866 to J. G. Holliday; 59348 of Nov. 6, 1866 to J. F. Boynton; 59462 of Nov. 6, 1866 to H. K. Schauck; 60957 of Jan. 1, 1867 to R. B. Stevenson; 62115 of Feb. 19, 1867 to J. P. Cowing; 69889 of Oct. 15, 1867 to C. B. Allen; 100167 of Feb. 22, 1870 to P. N. Mackay; 181528 of Aug. 29, 1876 to C. L. Fowler; 267045 of Nov. 7, 1882 to R. S. Waring and J. B. Hyde; 267046 of Nov. 7, 1882 to R. S. Waring; Reissue 10350 of Jul. 3, 1883 to R. S. Waring and J. B. Hyde; Reissue 10351 of Jul. 3, 1883 to R. S. Waring; 801111 of Oct. 3, 1905 to A. E. Schutté; 861294 of Jul. 30, 1907 to M. P. Lanza; 1320709 of Nov. 4, 1919 to M. A. Popkess; **Can. Pats.** 54681 of Jan. 20, 1897 to Terrence Sparham and James Thompson; 120730 of Sep. 21, 1909 to D. S. Mitchell; **Brit. Pats.** of 1881 (Jun. 27), 2185 to A. M. Clark; of 1906 (Sep. 26), 21290 to J. Johnson and J. Titley; of 1907 (Apr. 23), 9375 to H. S. Flint; of 1907 (May 8) 10699 to T. Marriott; of 1911 (Jul. 12), 16125 to T. Marriott; 372754 and 372755 of Feb. 10, 1931 to A. Wolf.

53 [p. 498] "Fuller's Earth," by C. L. Parsons, Bull. No. 71, Mineral Technology No. 3, Dept. of Interior, Bureau of Mines, Wash., D. C. (1913); "New Uses for Spent Fuller's Earth," by H. L. Kauffman, *Natl. Petrol. News*, **27**, 25 (1935); **U. S. Pat.** 1279918 of Sep. 24, 1918 to Wallace Savage; **Brit. Pat.** 424415 of Jul. 17, 1933 to Rudolph Hermann, Ltd.; **Ger. Pat.** Appl. B–451.30 of Nov. 15, 1930 to J. A. Braun.

54 [p. 498] **U. S. Pats.** 46495 of Feb. 21, 1865 to W. L. Potter; 85687 of Jan. 5, 1869 to John Munn; 90084 of May 18, 1869 to J. E. Dotch and Edward Duempelman; 107756 of Sep. 27, 1870 to Charles Burgess; 128599 of Jul. 2, 1872 to W. B. Davies; 148396 of Mar. 10, 1874 to C. W. Wilson; 246359 of Aug. 30, 1881 to W. B. Allman; 803190 of Oct. 31, 1905 to C. H. Parker and John Mandile; 910989 of Jan. 26, 1909 to P. J. Cannon, W. H. Young and Clinton Bateholts; 1548763 of Aug. 4, 1925 to G. L. Sleight; **Brit. Pats.** of 1876 (Dec. 23), 4966 to O. Brooke; of 1891 (Mar. 17), 4750 to Soc. anom. des Ardoisières de Deville and V. van der Heyden.

55 [p. 498] **U. S. Pats.** 40649 of Nov. 17, 1863 to Abraham Straub; 44675 of Oct. 11, 1864 to Isaac Straub; 61746 of Feb. 5, 1867 to R. O. Lowrey; 63803 of Apr. 16, 1867 to R. O. Lowrey; 64433 of May 7, 1867 to R. O. Lowrey; 76276 of Mar. 31, 1868 to W. P. Valentine; 72977 of Jan. 7, 1868 to A. Cody, James Bartlett and H. M. Jones; 771173 of Dec. 13, 1904 to E. I. Allison; **Brit. Pat.** 417696 of Oct. 10, 1934 to T. McQ. Swan.

56 [p. 498] **Brit. Pat.** 399176 of Sep. 26, 1933 to Crocker, King & Co.

57 [p. 498] **French Pats.** 579354 of Jun. 14, 1923 to C. Le Bos d'Espinoy; 732142 of Feb. 20, 1932 to L. C. Abraham and Rudolf Schmidtmann.

58 [p. 498] **Ger. Pat.** 605234 of Nov. 7, 1934 to Chemieprodukte, G.m.b.H.

59 [p. 498] **U. S. Pat.** 62810 of Mar. 12, 1867 to A. T. Boon; 748270 of Dec. 29, 1903 to W. O. Emery.

60 [p. 498] **U. S. Pats.** 24105 of May 24, 1859 to J. M. Day and E. H. A. Oakley; 28070 of May 1, 1860 to Charles Fricke; 44938 of Nov. 8, 1864 to Joseph Clarke; 57324 of Aug. 21, 1866 to Benjamin Hinkley; 59713 of Nov. 13, 1866 to Louis de l'Homme and Angelo Lazzaro; 65258 of May 28, 1867 to William Myler; 73645 of Jan. 21, 1868 to S. B. Pierce and Pembroke Pierce; 76276 of Mar. 31, 1868 to W. P. Valentine; 110343 of Dec. 20, 1870 to J. H. Cole; 124117 of Feb. 27, 1872 to D. G. Conger; 126026 of Apr. 23, 1872 to D. G. Conger; 352445 of Nov. 9, 1886 to J. W. Butler; 667786 of Feb. 12, 1901 to F. A. Lobert; 777173 of Dec. 13, 1904 to E. I. Allison; 822602 of Jun. 5, 1906 to August Gross and A. C. Horn; 1329232 of Jan. 27, 1920 to W. C. Erler; 1374885 of Apr. 12, 1921 to C. S. Hathaway; **Can. Pats.** 2132 of Mar. 7, 1873 to D. G. Conger; 6902 of Dec. 28, 1876 to Joseph Cérat; **Brit. Pats.** of 1899 (May 11), 9966 to J. Jungbluth; 368800 of Nov. 5, 1930 to Hermann Plauson and Maurice

Imer; **Ger. Pat.** 98071 of Mar. 4, 1897 to Chem. Fabrik Zimmer & Gottstein; **French Pats.** 794524 of Feb. 19, 1936 to "Straba" Strassenbaubedarfs A.-G.; 762597 of Apr. 13, 1934 to Emile Ingold.

61 [p. 498] **Ger. Pat.** 563360 of Nov. 20, 1928 to Iván Kardos.

62 [p. 498] **Brit. Pat.** 369481 of Jun. 19, 1931 to D. J. Pickée.

63 [p. 498] "Talc and Soapstone; their Mining, Milling, Products and Uses," by R. B. Ladoo, Bull. No. 213, Bureau of Mines, Dept. of Interior, Wash., D. C. (1923); **U. S. Pats.** 65258 of May 28, 1867 to William Myler; 93245 of Aug. 3, 1869 to L. S. Stimson; 107756 of Sep. 27, 1870 to Charles Burgess; 117601 of Aug. 1, 1871 to C. W. Chaffee; 159629 of Feb. 9, 1875 to A. N. Atwood; 168142 of Sep. 28, 1875 to J. A. Craig; 278536 of May 29, 1883 to John Greives; 282139 of Jul. 31, 1883 to Welcome White; 389050 of Sep. 4, 1888 to C. H. Bushnell; 791960 of Jun. 6, 1905 to F. J. Warren; **Can. Pats.** 131756 of Mar. 21, 1911 to E. E. Blackburn; 324251 of Jul. 20, 1932 to William Hassard.

64 [p. 498] **U. S. Pats.** 214091 of Apr. 8, 1879 to J. B. Bowen; 282139 of Jul. 31, 1883 to Welcome White; 1045585 of Nov. 26, 1912 to W. J. Moeller; 1796474 of Mar. 17, 1931 to G. A. Osterday; 1801245 of Apr. 14, 1931 to E. L. Chamberlain; **Can. Pat.** 69016 of Oct. 16, 1900 to James Cunningham.

65 [p. 498] "Bitumen in Vermischung mit Mineralmehlen," by Heinrich Rosenberg, *Bitumen*, **9**, 298 (1932); "Ueberwindung der Sprödigkeit des Asphalts durch Teerzusatz oder durch zusatz von Microasbest," by Walter Obst, *Teer u. Bitumen*, **30**, 383 (1932); "Microasbestos in the Asphalt Industry and Street Construction," by Heinrich Rosenberg, *Petroleum Z.*, **29**, 1 (1933).

66 [p. 498] **U. S. Pats.** 320979 of Jun. 30, 1885 to L. B. Snow; 1237000 of Aug. 14, 1917 to Herman von Uffel; 1773131 of Aug. 19, 1931 to C. R. Eckert; 2009811 and 2009812 of Jul. 30, 1935 to Sigurd Olsen; 2047772 of Jul. 14, 1936 to C. R. Eckert; **Can. Pats.** 16060 of Jan. 2, 1883 to W. L. Maltby; 16175 of Jan. 23, 1883 to W. L. Maltby; 17102 of Jun. 18, 1883 to W. L. Maltby; 44319 of Sep. 20, 1893 to S. D. Chatterton; 95008 of Sep. 5, 1905 to R. H. Walker; 308508 of Feb. 10, 1931 to Barrett Co.; 356104 of Feb. 25, 1936 to Sigurd Olsen; **Brit. Pats.** of 1891 (Jun. 3), 9434 to John Menzies; 343265 of Jan. 1, 1930 to Barrett Co.; **Ger. Pat.** Appl. B–258.30 of Jul. 22, 1930 to Barrett Co.; 613620 of May 23, 1935 to International Bitumen Emulsions Corp.; **Austrian Pat.** 55651 of Nov. 1, 1913 to H. P. Rasmussen.

67 [p. 498] **Brit. Pat.** 434926 of Mar. 26, 1935 to Planktokoll Chem. Fabrik, G.m.b.H.; **Ger. Pat.** 631900 of Mar. 23, 1935 to Planktokoll Chem. Fabrik G.m.b.H.; **Belgian Pat.** 408393 of Apr. 30, 1935 to A. Esser and B. Fisel.

68 [p. 498] **U. S. Pats.** 1008433 of Nov. 14, 1911 to M. A. Popkess; 1220680 and 1220681 of Mar. 27, 1917 to M. A. Popkess; 1227903 of May 29, 1917 to G. C. Glynn; 1240481 of Sep. 18, 1917 to M. A. Popkess; 1252722 of Jan. 8, 1918 to George Ross; 1278663 of Sep. 10, 1918 to C. R. Hunt; 1327726 of Jan. 13, 1920 to S. R. Murray; 1330443 of Feb. 10, 1920 to G. C. Glynn; 1812730 of Jun. 30, 1931 to W. C. West; **Brit. Pats.** 351385 of Dec. 17, 1929 to H. R. Seabrook; 376792 of Jul. 13, 1932 to William Griffiths; 399176 of Mar. 23, 1932 to R. H. King and A. O. Crocker; 441503 of Jan. 21, 1936 to W. J. Woodfine.

69 [p. 498] **U. S. Pats.** 59682 of Nov. 13, 1866 to J. P. Thompson; 59713 of Nov. 13, 1866 to Louis de l'Homme and Angelo Lazzaro; 70478 of Nov. 5, 1867 to G. O. Smith and J. H. Smith; 71667 of Dec. 3, 1867 to Reuben Tyler and Peter Campbell, Jr.; 142595 of Sep. 9, 1873 to Albert Thiele; 206175 of Jul. 23, 1878 to E. W. Hickman; 267046 of Nov. 7, 1882 to R. S. Waring; 278536 of May 29, 1883 to John Greives; Reissue 10351 of Jul. 3, 1883 to R. S. Waring; 282914 of Aug. 7, 1883 to J. F. Martin; 291718 of Jan. 8, 1884 to John Greives; 334782 of Jan. 26, 1886 to Fred Kimble; 667786 of Feb. 12, 1901 to F. A. Lobert; 1771795 of Jul. 29, 1930 to T. F. King; **Can. Pat.** 336064 of Oct. 3, 1933 to T. F. King; **Brit. Pats.** 158674 of Nov. 7, 1919 to Maurice Talbot-Crosbie; 260621 of Oct. 30, 1926 to H. H. Schröder; 398268 of Sep. 14, 1933 to E. W. Harvey and H. F. Weatherly; **Ger. Pats.** 96094 of Mar. 11 1896 to Richard and Johannes Joecks; 104194 of Feb. 17, 1898 to M. Rauschenbusch; 189069 of Sep. 2, 1904 to C. F. Lau; 279581 of Dec. 23, 1913 to Wilhelm Reppin; 289610 of Jun. 3 1914 to Wilhelm Reppin; 305789 of Jun. 29, 1916 to Cooper & Co.; 402731 of Dec. 11, 1921 to K. Wehle.

70 [p. 498] **U. S. Pats.** 1164041 of Dec. 14, 1915 to G. C. Warren; 1235270 of Jul. 31, 1917 to J. B. Wise; 1926630 of Sep. 9, 1933 to W. R. Parker and W. L. Holbrook; 2056520 of Oct. 6, 1936 to W. L. Holbrook and W. R. Parker; 2063893 of Dec. 15, 1936 to W. L. Holbrook and W. R. Parker.

71 [p. 498] **Ger. Pat.** 107947 of Feb. 15, 1899 to Otto Pötzsch.

72 [p. 498] **U. S. Pat.** 84120 of Nov. 17, 1868 to Dwight Hitchcock.

73 [p. 498] **U. S. Pats.** 572645 of Dec. 8, 1896 to St. Vrain le Sieur; 1418905 of Jun. 6, 1922 to K. L. Binkley; **Ger. Pat.** 64680 of Sep. 23, 1891 to Bruno Roedelius.

74 [p. 498] **U. S. Pat.** 56818 of Jul. 31, 1866 to R. B. Smith.

75 [p. 498] **U. S. Pats.** 24105 of May 24, 1859 to J. M. Day and E. H. A. Oakley; 34200 of Jan. 21, 1862 to John Duke; 42925 of May 31, 1864 to Stephen Bowerman; 44938 of Nov. 8, 1864 to Joseph Clarke; 57400 of Aug. 21, 1866 to C. Spear; 69889 of Oct. 15, 1867 to C. B. Allen; 75634 of Mar. 17, 1868 to J. H. Hood; 93482 of Aug. 10, 1869 to S. I. Russell and J. H. Cole; 110343 of Dec. 20, 1870 to J. H. Cole; 124117 of Feb. 27, 1872 to D. G. Conger; 126026 of Apr. 23, 1872 to D. G. Conger; 206175 of Jul. 23, 1878 to E. W. Hickman; 334782 of Jan. 26, 1886 to Fred Kimble; 393644 of Nov. 27, 1888 to Salomon Heimann; 803190 of Oct. 31, 1905 to C. H. Parker and John Mandile; **Can. Pats.** 2132 of Mar. 7, 1873 to D. G. Conger; 6902 of Dec. 28, 1876 to Joseph Cérat; **Brit. Pat.** 302767 of Oct. 26, 1927 to E. C. R. Marks; **Ger. Pats.** 20885 of Apr. 16, 1882 to C. Brasche and L. Mitgau; 576768 of May 16, 1933 to E. R. Taylor.

76 [p. 498] **Ger. Pat.** 95884 of Jun. 24, 1896 to Lebach & Co.

77 [p. 498] **U. S. Pat.** 1774608 of Sep. 2, 1930 to H. L. Mead.

78 [p. 498] **U. S. Pats.** 52973 of Mar. 6, 1866 to Edward Curtis and Andrew Crozier; 59713 of Nov. 3, 1866 to Louis de l'Homme and Angelo Lazzaro; 95071 of Sep. 21, 1869 to Giacinto Bartolmei.

79 [p. 498] **U. S. Pats.** 1220682 of Mar. 27, 1917 to M. A. Popkess; 1364622 of Jan. 4, 1921 to J. R. Draney and L. M. Law; **Can. Pat.** 210186 of Apr. 5, 1921 to Bitoslag Paving Co.; **Brit. Pat.** 169079 of Jul. 30, 1920 to W. J. Mellersh-Jackson.

80 [p. 498] **U. S. Pats.** 34543 of Feb. 25, 1862 to W. H. White; 34653 of Mar. 11, 1862 to Zadok Street; 67998 of Aug. 20, 1867 to A. H. Mott, Daniel Winer and Lawrence Brink; 89186 of Apr. 20, 1869 to C. G. Von Tagen; 121118 of Nov. 21, 1871 to James McKenzie and J. M. Stebbins; 152503 of Jun. 30, 1874 to John McDerby and A. G Stevens; 162503 of Apr. 27, 1875 to Basile St. Jean; 298072 of May 6, 1884 to D. H. Dorsett; 1051769 of Jan. 28, 1913 to F. M. Ruschhaupt; 1237921 of Aug. 21, 1917 to E. J. Lovegrove and N. G. Crompton; 1352931 of Sep. 14, 1920 to Oscar Altpeter; 1369911 of Mar. 1, 1921 to T. H. Brown and Haughton Brown; 1370990 of Mar. 8, 1921 to B. W. O'Connell and Dennis O'Connell; **Can. Pat.** 8418 of Feb. 12, 1878 to John Brokenshire; **Brit. Pats.** of 1875 (Jun. 9), 4267 to Alexander Wilkinson; 361564 of Oct. 30, 1930 to Rudolf Traut.

81 [p. 498] **Brit. Pat.** 361564 of Oct. 30, 1930 to Rudolf Traut; **Ger. Pat.** Appl. D–285.30 of Jul. 29, 1930 to Ludwig Dreibholz; 622604 of Oct. 31, 1933 to Wigankow & Sachse Neuzeitlicher Strassenbau G.m.b.H.

82 [p. 498] **U. S. Pats.** 81838 of Apr. 3, 1868 to Benjamin Stephens; 170132 of Nov. 16, 1875 to J. C. Tucker; 1211382 of Jan. 9, 1917 to Oscar Altpeter; 1364621 of Jan. 4, 1921 to J. R. Draney; 1884471 of Oct. 25, 1932 to Lester Wittenberg; **Can. Pat.** 8418 of Feb. 12, 1878 to John Brokenshire; **Brit. Pat.** 167997 of Dec. 23, 1920 to Bitoslag Paving Co.; **Ger. Pats.** 527934 of Oct. 5, 1927 to Musag Ges. für den Bau von Müll- und Schlacken- Verwertungsanlagen, A.-G.; 554221 of Jul. 28, 1929 to Oskar Dönig; **Austrian Pat.** of 1887 (Jul. 5), 225 to Louis Baffrey.

83 [p. 498] **U. S. Pats.** 72977 of Jan. 7, 1868 to A. Cody, James Bartlett and H. M. Jones; 90084 of May 18, 1869 to J. E. Dotch and Edward Duempelman; 932622 of Aug. 31, 1909 to T. D. Lynch and J. J. Crane.

84 [p. 498] **U. S. Pats.** 46975 of Mar. 21, 1865 to C. G. Reinhold; 74606 of Feb. 18, 1868 to H. K. Schanck; 80084 of Jul. 21, 1868 to J. A. Moore; 169385 of Nov. 2, 1875 to L. P. Teed; 320979 of Jun. 30, 1885 to L. B. Snow; 393644 of Nov. 27, 1888 to Soloman Heimann; 686191 of Nov. 5, 1901 to W. H. Bache; 853117 of May 7, 1907 to Clifford Richardson and

C. N. Forrest; **Brit. Pats.** of 1884 (May 28), 8332 to Jules Hautrive; of 1887 (Sep. 17), 12632 to J. C. Lyman; of 1891 (Jun. 3), 9434 to John Menzies; **Ger. Pats.** 83096 of Jan. 16, 1894 to Carl Jost; 183552 of Nov. 18, 1904 to Hans Christen; **Austrian Pat.** 41300 of Mar. 10, 1910 to Emil Kuznitzki; **Japan Pat.** 35965 of Mar. 11, 1920 to Y. Sakakibara.

85 [p. 498] **U. S. Pat.** 1559731 of Nov. 3, 1925 to G. J. Muller.

86 [p. 498] **U. S. Pat.** 1719118 of Jul. 2, 1929 to A. R. Lukens and J. S. Maynard.

87 [p. 499] **U. S. Pats.** 142130 of Aug. 26, 1873 to J. B. Schlichter; 221502 of Nov. 11, 1879 to P. A. Bell; 276891 of May 1, 1873 to J. G. Sanderson; **Ger. Pat.** 357611 of Aug. 26, 1918 to A. Blumenthal & Co., Chemische Fabrik.

88 [p. 499] **Ger. Pat.** 163002 of Sep. 13, 1905 to F. C. Matthies et Cie.

89 [p. 499] **U. S. Pats.** 128599 of Jul. 2, 1872 to W. B. Davies; 221502 of Nov. 11, 1879 to P. A. Bell; **Brit. Pat.** of 1888 (Mar. 5), 3354 to Thomas Thomson.

90 [p. 499] **Ger. Pat.** 368236 of Dec. 24, 1920 to Lack- und Farbenfabriken Max Rogler.

91 [p. 499] "New Packing Material (Sinterite) for Socket Joints," by H. Vogt, *Gas- und Wasserfach*, **79**, 592 (1936); **U. S. Pats.** 19627 of Mar. 16, 1858 to W. T. de Golyer; 47416 of Apr. 25, 1865 to Nicolaus Groh; 107232 of Sep. 13, 1870 to J. V. Douglass; 1465107 of Aug. 14, 1923 to Katharina Wickel; **Can. Pat.** 80476 of Apr. 21, 1903 to W. S. Wilkinson; **Brit. Pats.** of 1895 (Dec. 20), 24479 to R. D. Upham; of 1898 (Oct. 11), 21346 to Clemens Dörr; of 1913 (Nov. 14), 26173 to J. H. Francis; 183143 of Jul. 17, 1922 to Katharina Wickel; 435732 of Sep. 26, 1935 to N. V. de Bataafsche Petroleum Maatschappij; **Ger. Pat.** Appl. T–38898 of May 21, 1931 to Ernst Täuber.

92 [p. 499] **U. S. Pats.** 79732 of Jul. 7, 1868 to F. A. Curtis; 447412 of Mar. 3, 1891 to J. S. Palmer; 1329232 of Jan. 27, 1920 to W. C. Erler; **Ger. Pat.** 215682 of Sep. 5, 1907 to Ludwig Grote.

93 [p. 499] **Brit. Pat.** of 1912 (Feb. 19), 4148 to J. Radcliffe.

94 [p. 499] **U. S. Pat.** 69618 of Oct. 8, 1867 to A. T. Boon and Joseph Stafford.

95 [p. 499] **Brit. Pat.** 424494 of Feb. 14, 1935 to Maurice Ernotte.

96 [p. 499] **U. S. Pats.** 226547 of Apr. 13, 1880 to J. L. Pope; 452182 of May 12, 1891 to F. C. Goodall; 460249 of Sep. 29, 1891 to R. F. Flynn; 674219 of May 14, 1901 to J. A. Scharwath; 1796474 of Mar. 17, 1931 to G. A. Osterday; **Can. Pat.** 364137 of Feb. 16, 1937 to Barrett Co.; **Brit. Pat.** of 1891 (Jun. 3), 9434 to John Menzies; **French Pat.** 762692 of Apr. 16, 1934 to André Léauté.

97 [p. 499] **U. S. Pat.** 99267 of Jan. 25, 1870 to P. Werni.

98 [p. 499] **U. S. Pats.** 23248 of Mar. 15, 1859 to John Hobrecker; 790932 of May 30, 1905 to J. B. Swindle; **Can. Pat.** 359242 of Jul. 21, 1936 to De jydske Skaerverfabriker; **Brit. Pats.** 446540 of Jan. 23, 1935 to Robert Kuftner; 448814 of May 11, 1935 to De jydske Skaerverfabriker; **Ger. Pat.** 631289 of Jun. 17, 1936 to Gesellschaft für Teerstrassenbau m.b.H.

99 [p. 499] **French Pat.** 749094 of Jul. 18, 1933 to Enterprises Albert Cochery, S. A.

100 [p. 499] "Ageing of Coal-tar Coatings," by André Léauté, *Compt. rend.*, **197**, 751, (1933); **U. S. Pats.** 62409 of Feb. 26, 1867 to David Green; 401014 of Apr. 9, 1889 to Alfonso de Figanière; 565675 of Aug. 11, 1896 to E. T. Dumble; 1961678 of Jun. 5, 1934 to J. T. Sweeney; **Brit. Pats.** of 1875 (Jun. 9), 4267 to Alexander Wilkinson; of 1908 (Mar. 4), 4947 to V. A. Noodt and G. Göttsche; 341901 of Jul. 23, 1929 to South Metropolitan Gas Co.; 431474 of Jul. 8, 1935 to Soc. de Recherches et de Perfectionnements Industriels; **Ger. Pat.** Design 1277629 of Sep. 7, 1933 to Baeumer & Loesch.

101 [p. 499] **U. S. Pat.** 103169 of May 17, 1870 to J. L. Fulton and Julius Brace; **Can. Pats.** 351222 of Jun. 25, 1935 to Standard Oil Development Co.; 358153 of May 26, 1936 to Colprovia Roads, Inc.; **Brit. Pats.** 354433 of Oct. 14, 1929 to H. Milke and J. Oberbach; 375953 of May 22, 1931 to Baldry Yerburgh and W. Pohl; **Ger. Pats.** 28620 of Jan. 20, 1884 to Emil Dietrich; 332690 of May 30, 1918 to Heinrich Jansen; 512707 of Mar. 7, 1928 to Wilhelm Reiner; **French Pat.** 758243 of Jan. 12, 1934 to Barber Asphalt Co.

102 [p. 499] **Ger. Pat.** 598470 of Jan. 3, 1931 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger, A.-G.

103 [p. 499] **U. S. Pats.** 24105 of May 24, 1859 to J. M. Day and E. H. A. Oakley;

169385 of Nov. 2, 1875 to L. P. Teed; 425412 of Apr. 15, 1890 to I. T. Dyer; 727506 and 727507 of May 5, 1903 to F. J. Warren; 1864971 of Jun. 28, 1932 to J. H. Young and P. W. Jenkins; **Can. Pat.** 327880 of Nov. 22, 1932 to H. H. Robertson Co.; **Brit. Pats.** of 1902 (Apr. 22), 9322 to H. H. Lake; 392372 of Jun. 28, 1932 to J. H. Young and P. W. Jenkins.

104 [p. 499] **U. S. Pats.** 122962 of Jan. 23, 1872 to C. H. Pond; 227352 of May 11, 1880 to Edgar Everhart; **Brit. Pats.** of 1855 (Sep. 17), 2095 to E. Gibbs; 431781 of Jul. 8, 1935 to Soc. de Recherches et de Perfectionnements Industriels; **Fr. Pat.** 590379 of Oct. 23, 1924 to U. Chandeysson and L. Allibert; 762692 (Additions 44832 and 44839) of Apr. 15, 1935 to André Léauté.

105 [p. 499] **U. S. Pats.** 34653 of Mar. 11, 1862 to Zadok Street; 53281 of Mar. 20, 1866 to George Duncan; 58148 of Sep. 18, 1866 to Jesse Stow and James White; 70478 of Nov. 5, 1867 to G. O. Smith and J. H. Smith; 78269 of May 26, 1868 to D. W. Denton; 91223 of Jun. 15, 1869 to John Fullagar and Miles Byrne; 547097 of Oct. 1, 1895 to Henry Van Newkirk; 547104 of Oct. 1, 1895 to J. C. Zallée; 2037147 of Apr. 14, 1936 to John Radcliffe; 2038023 of Apr. 21, 1936 to W. A. Craig and F. E. Griffith; **Brit. Pat.** of 1878 (May 9), 1865 to A. M. Gobin; 387324 of May 26, 1931 to J. Radcliffe; **Ger. Pats.** 125891 of Nov. 24, 1900 to Hans Christen; 238842 of Dec. 24, 1909 to S. J. Sauvageot; 604114 of Jul. 3, 1936 to Hans Wiedemann; **French Pat.** 579354 of Jun. 14, 1923 to C. Le Bos d'Espinoy.

106 [p. 499] **U. S. Pats.** 159629 of Feb. 9, 1875 to A. N. Atwood; 171209 of Dec. 21, 1875 to Alanson Bernard; 268034 of Nov. 28, 1882 to Murdoch Mackay; 282139 of Jul. 31, 1883 to Welcome White; 300464 of Jun. 17, 1884 to Levi Haas; 315633 of Apr. 14, 1885 to Henry Kellogg; 319084 of Jun. 2, 1885 to J. A. Fleming; 320979 of Jun. 30, 1885 to L. B. Snow; 429999 of Jun. 10, 1890 to C. A. Catlin; 571117 of Nov. 10, 1896 to F. R. Hall; 580139 of Apr. 6, 1897 to William Morison; 910989 of Jan. 26, 1909 to P. J. Cannon, W. H. Young and Clinton Bateholts; 939982 of Nov. 16, 1909 to F. J. Conboy; 1025268 of May 7, 1912 to Emile Hemming; 1211382 of Jan. 9, 1917 to Oscar Altpeter; 1304483 of May 20, 1919 to Heisaburo Inoue; 1374885 of Apr. 12, 1921 to C. S. Hathaway; 1516469 of Nov. 18, 1924 to J. S. Miller, Jr.; 1517360 of Dec. 2, 1924 to F. J. Groten, Jr.; 1901792 of Mar. 14, 1933 to Georg Zimmermann; 1943085 of Jan. 9, 1934 to Karl Müller and Eugen Hutzenlaub; 2054285 of Sep. 15, 1936 to Benjamin Foster; **Can. Pats.** 16074 of Jan. 5, 1883 to Charles Taylor, Joseph James and Enoch James; 16212 of Jan. 25, 1883 to W. L. Maltby; 17565 of Sep. 1, 1883 to Welcome White; 31049 of Apr. 8, 1889 to F. T. Tinning; 106042 of Jun. 25, 1907 to Robert Müller; 208341 of Feb. 8, 1921 to B. W. Jacobs; 234293 of Sep. 18, 1923 to Steven Troy; 337340 of Nov. 21, 1933 to Industriel Processes Development, Ltd.; **Brit. Pats.** of 1884 (Apr. 22), 6647 to J. A. Fleming; of 1896 (Aug. 20), 18485 to William Morison; of 1897 (Apr. 12), 9290 to William Morison; of 1907 (Oct. 19), 23093 to E. Müller; of 1907 (Oct. 31), 24052 to Robert Müller; of 1910 (May 13), 11908 to A. R. Müller; of 1915 (May 17), 7384 to A. A. Lockwood; 116532 of Jun. 11, 1917 to A. R. Müller; **Ger. Pat.** 167166 of Jan. 12, 1906 to Robert Müller; **Fr. Pat.** 429576 of May 10, 1911 to A. R. Müller; **Austrian Pat.** 4290 of May 25, 1901 to G. Lesigang.

107 [p. 499] **Brit. Pats.** 393270 of Oct. 30, 1931 to Carl Alfeis; 406505 of Apr. 20, 1933 to Carl Alfeis; **Ger. Pats.** 95884 of Jun. 24, 1896 to Lebach & Co.; 531861 of Jan. 27, 1929 to Carl Alfeis; 556702 of Jan. 27, 1929 to Carl Alfeis; 571699 of Apr. 21, 1932 to Carl Alfeis; 592047 of May 3, 1933 to Carl Alfeis; 596725 of May 14, 1934 to Ruberoidwerke, A.-G.; Appl. R–80559 of Jan. 30, 1931 to Paul Lewino (Ruberoidwerke, A.-G.); **French Pat.** 758370 of Jan. 15, 1934 to Carl Alfeis; **Austrian Pat.** 136008 of Dec. 27, 1933 to Franz Jaray.

108 [p. 499] "High-quality Bituminous Calking Material with Asbestos Filler and the Preparation of Substitutes with German Raw Materials," by P. Herrmann, *Asphalt Teer Strassenbautech.*, **35**, 206 (1935); "Anregungen zur Zusammensetzung und Prüfung bituminöser Betonfugenvergussmassen," by Hans Nüssel, *Bitumen*, **6**, 99 (1936); "Zur Frage der Herstellung bituminöser Vergussmassen für Betonfugen," by W. Becker, *Bitumen*, **6**, 173 (1936); "Auswertung von Versuchen mit Bitumenvergussmassen für Betonfugen," by R. Sack, *Bitumen*, **7**, 7 (1937); **U. S. Pats.** 319084 of Jun. 2, 1885 to J. A. Fleming; 351611 of Oct. 26, 1886 to Robert Alexander; 452763 of May 19, 1891 to Frederick Salathé; 1357920

of Nov. 2, 1920 to Herbert Abraham; **Brit. Pat.** of 1904 (Mar. 8), 5648 to J. H. W. Stringfellow; **Ger. Pats.** 331675 of Sep. 9, 1919 to H. Brunck; 360078 of Mar. 8, 1921 to Herbert Abraham.

109 [p. 499] **U. S. Pats.** 351611 of Oct. 28, 1886 to R. Alexander; 1824430 of Sep. 22, 1931 to C. C. Hall; 1996082 of Apr. 2, 1935 to E. R. Powell; **Can. Pat.** 317381 of Nov. 24, 1931 to E. R. Powell

110 [p. 499] **U. S. Pats.** 75634 of Mar. 17, 1868 to J. H. Hood; 103536 of May 31, 1870 to T. R. Abbott; 162503 of Apr. 27, 1875 to Basile St. Jean; 268034 of Nov. 28, 1882 to Murdoch Mackay; 278481 of May 29, 1883 to S. M. Allen; 284289 of Sep. 4, 1883 to J. A. Fleming; 284794 of Sep. 11, 1883 to S. M. Allen; 300464 of Jun. 17, 1884 to Levi Haas; 319084 of Jun. 2, 1885 to J. A. Fleming; 337472 of Mar. 9, 1886 to S. M. Allen; 447412 of Mar. 3, 1891 to J. S. Palmer; 1187314 of Jun. 13, 1916 to G. A. Henderson; 1203403 of Oct. 31, 1916 to Mathias Poulsen; 1370647 of Mar. 8, 1921 to G. A. Henderson; 1372640 of Mar. 22, 1921 to A. L. Clapp; 1374187 of Apr. 12, 1921 to A. L. Clapp; 1604728 of Oct. 26, 1926 to D. M. Sutherland, Jr.; 2072686 and 2072687 of Mar. 2, 1937 to Thomas Robinson; **Can. Pat.** 228285 of Jan. 23, 1923 to J. W. Tucker; **Brit. Pats.** of 1863 (Jun. 25), 1603 to William Kirrage; of 1872 (Jul. 9), 2065 to W. R. Lake; of 1874 (Mar. 4), 790 to F. Wirth; of 1875 (Jun. 9), 4267 to Alexander Wilkinson; of 1876 (Apr. 26), 1750 to J. Noad; of 1880 (Jan. 9), 94 to Richard Condy; of 1894 (Mar. 8), 4914 to Anton Pieper; of 1897 (Apr. 23), 10181 to M. van Look; of 1911 (Feb. 23), 4589 to J. Radcliffe; of 1911 (Sep. 27), 20075 to Chem. Fabrik Lindenhof, C. Weyl & Co.; of 1912 (Apr. 6), 8341 to G. A. Henderson; 112077 of Apr. 3, 1917 to G. H. Culling; 125492 of Apr. 12, 1918 to S. G. Kelsey; 134538 of Oct. 29, 1918 to I. T. Dyring; 148641 of Apr. 30, 1919 to G. A. Henderson; **Ger. Pats.** 23780 of Nov. 12, 1882 to Hermann Klette; 253377 of Jul 19, 1911 to Albin Baer; 368298 of May 11, 1920 to Rütgerswerke A.-G. and H. Teichmann; Appl. C–50227 of Feb. 22, 1935 to Chemische Fabrik Grünau Landshoff & Meyer; **Fr. Pat.** 454082 of Feb. 7, 1913 to Denniel & Cie.

111 [p. 499] **U. S. Pats.** 278481 of May 29, 1883 to S. M. Allen; 372552 of Nov. 1, 1887 to Terrence McSweeney; 452763 of May 19, 1891 to Frederick Salathé; 1357920 of Nov. 2, 1920 to Herbert Abraham; **Brit. Pat.** of 1911 (Apr. 6) 8622 to W. H. Rymer.

112 [p. 499] **Ger. Pat.** 165342 of Feb. 20, 1904 to B. Holczabek.

113 [p. 499] **U. S. Pat.** 1369911 of Mar. 1, 1921 to T. H. Brown and Haughton Brown; **Brit. Pat.** 451192 of Nov. 11, 1935 to T. C. Thomsen; **French Pat.** 797468 of Apr. 27, 1936 to Holback Tagpap Og Cementvarefabrikker A/S.

114 [p. 499] **U. S. Pat.** 108334 of Oct. 18, 1870 to D. W. Denton; **French Pat.** 389433 of Jun. 28, 1907 to Edmond Barthelet.

115 [p. 499] **U. S. Pats.** 172321 of Jan. 18, 1876 to C. B. Hutchins; 214091 of Apr. 8, 1879 to J. B. Bowen; 319084 of Jun. 2, 1885 to J. A. Fleming.

116 [p. 499] **U. S. Pat.** 1593071 of Jul. 20, 1926 to R. J. Gruenberg; **Brit. Pat.** of 1863 (Jun. 25), 1603 to William Kirrage.

117 [p. 499] **Brit. Pat.** 102826 of Jan. 7, 1916 to J. E. Clark.

118 [p. 499] **Brit. Pats.** of 1872 (Apr. 27), 1264 to W. A. Lyttle; of 1905 (Jun. 6), 11859 to T. Marriott and A. Darch; of 1909 (Dec. 7), 28547 to L. F. von Sachs; **Fr. Pat.** 389433 of Jun. 29, 1908 to Edmond Barthelet.

119 [p. 499] **U. S. Pats.** 68106 of Aug. 27, 1867 to J. D. Numan, J. T. Wilkinson and E. W. Cook; 108666 of Oct. 8, 1870 to J. T. Wilkinson; 254461, 254462 and 254463 of Mar. 7, 1882 to J. D. Cheever; 352445 of Nov. 9, 1886 to J. W. Butler; **Brit. Pats.** of 1863 (Jan. 27), 232 to H. H. Henson; of 1872 (Apr. 27), 1264 to W. A. Lyttle; of 1875 (Jun. 9), 4267 to Alexander Wilkinson; of 1880 (Jan. 9), 94 to Richard Condy.

120 [p. 499] **U. S. Pats.** 172321 of Jan. 18, 1876 to C. B. Hutchins; 246359 of Aug. 30, 1881 to W. B. Allman; **Brit. Pat.** 114527 of Jun. 11, 1917 to A. R. Scarlett.

121 [p. 499] **U. S. Pat.** 300464 of Jun. 17, 1884 to Levi Haas; **Ger. Pats.** 293871 of Sep. 1, 1914 to Wilhelm Reiner; 294050 of Feb. 13, 1916 to Wilhelm Reiner.

122 [p. 499] **U. S. Pats.** 57400 of Aug. 21, 1866 to C. Spear; 60830 of Jan. 1, 1867 to J. F. Boynton; 290058 of Dec. 11, 1883 to J. B. Hyde; 393644 of Nov. 27, 1888 to Salomon Heimann; 515192 of Feb. 20, 1894 to G. A. Cannot; **Can. Pat.** 121045 of Oct. 5, 1909 to F. A.

Lallemand; **Brit. Pats.** of 1861 (Aug. 19), 2058 to W. H. Smith; of 1866 (Mar. 15), 779 to T. G. Ghislin; of 1872 (Apr. 27), 1264 to W. A. Lyttle; of 1881 (Jul. 28), 3279 to H. Armstrong and J. A. London; of 1881 (Oct. 22), 4628 to Alexander Wilkinson; of 1894 (Mar. 8), 4914 to Anton Pieper; 206266 of Aug. 14, 1922 to Gustav Pfarrius; **Ger. Pats.** 145251 of Oct. 5, 1902 to Factonit-Werke, Reif & Co., G.m.b.H.; 299386 of Sep. 1, 1915 to Gesellschaft für Torf-Isolation, m.b.H. **French Pat.** 791864 of Dec. 18, 1935 to De Jydske Skaervefabriker.

123 [p. 499] **U. S. Pat.** 2075751 of Mar. 30, 1937 to R. W. B. Reade and H. L. Levin; **Can. Pat.** 330054 of Feb. 7, 1933 to Patent & Licensing Corp.

124 [p. 500] "Fortschritte im Städte- und Strassenbau," by Hermann Ehlgötz and Karl Krüger, Vol. 2 ("Ueber stabilisierende Einfüsse von künstlichen und natürlichen Füllerbeimengungen auf Bitumen," by Paul Weichert), Allgemeiner Industrie-Verlag, G.m.b.H., Berlin (1929); "Der Einfluss staubförmiger Füllerstoffe auf die plastischen und elastischen Eigenschaften bituminöser Massen," by Hermann Suida and A. Schmölzer, *Petroleum Z.*, **25**, 251 (1929); "Zum Problem der talkumierten Teerdachpappe," by H. Walther, *Teer u. Bitumen*, **29**, 429 (1931); "Physical Properties of Mixtures of Bitumen and Finely Divided Mineral Matter," by A. Evans, *J. Inst. Petroleum Techn.*, **18**, 957 (1932); "Neue Wege für den Aufbau von Asphalt- und Teerdecken," by R. Wilhelmi, Allgemeiner Industrie-Verlag, G.m.b.H., Berlin (1933).

125 [p. 501] "The Properties of Various Kinds of Stone Meal and their Use as Fillers in Tar-Concrete Masses," by Willi Drescher, *Asphalt Teer Strassenbautech.*, **32**, 294, 312 and 396 (1932).

CHAPTER XXV

1 [p. 505] "Rôle of the Emulsifier in Bituminous Emulsions," by A. W. Rick, *Teer u. Bitumen*, **32**, 113 (1934).

2 [p. 505] **U. S. Pats.** 1668136 of May 1, 1928 to C. A. Agthe; 1817958 of Aug. 11, 1931 to F. Zwilgmeyer; 1842877 of Jan. 26, 1932 to Karl Müller and Eugen Hutzenlaub; 1961971 of Jun. 5, 1934 to Lionel Ignace; **Can. Pat.** 218270 of May 2, 1922 to Process Engineers, Ltd.; **Brit. Pats.** 362430 of Sep. 1, 1930 to Paul Lechler; 407153 of Dec. 9, 1932 to D. G. Murdoch and W. V. Shannan; **Ger. Pats.** 405237 of Mar. 14, 1922 to C. A. Agthe; Appl. C–39752 of Apr. 29, 1927 to F. G. E. Champilou; Appl. P–57072 of Feb. 9, 1928 to Otto Röhm; Appl. L–81496 of Apr. 16, 1930 to Paul Lechler; **French Pat.** 563564 of Mar. 12, 1923 to C. A. Agthe.

3 [p. 505] **U. S. Pats.** 1384805 of Jul. 19, 1921 to D. J. McSwiney; 1525409 of Feb. 3, 1925 to Hippolyte Neveu; 1542035 of Jun. 16, 1925 to M. R. Cone; 1560826 of Nov. 10, 1925 to Lester Kirschbraun; Reissue 16328 of Apr. 20, 1926 to Lester Kirschbraun; 1600948 of Sep. 21, 1926 to G. B. Poore; 1603546 of Oct. 19, 1926 to Lester Kirschbraun; 1615303 of Jan. 25, 1927 to Lester Kirschbraun; 1636644 of Jul. 19, 1927 to Hippolyte Neveu; 1663652 of Mar. 27, 1928 to H. R. Gundlach; 1679475 of Aug. 7, 1928 to Lester Kirschbraun; 1722433 of Jul. 30, 1929 to Lester Kirschbraun; 1733496 of Oct. 29, 1929 to Lester Kirschbraun; 1757065 of May 6, 1930 to H. E. Warsop and F. W. Gough; 1774205 of Aug. 26, 1930 to C. R. MacDonald; 1787338 and 1787339 of Dec. 30, 1930 to A. L. Clapp; 1789320 of Jan. 20, 1931 to F. C. Overbury; 1789897 of Jan. 20, 1931 to M. G. Gregg; 1792067 of Feb. 10, 1931 to G. A. Brown; 1793957 of Feb. 24, 1931 to R. K. Painter; 1795226 of Mar. 3, 1931 to L. R. MacKenzie; 1804562 of May 12, 1931 to Lester Kirschbraun and F. C. Overbury; 1829722 of Oct. 27, 1931 to Lester Kirschbraun and F. C. Overbury; 1866503 and 1866504 of Jul. 5, 1932 to Lester Kirschbraun; 1884664 of Oct. 25, 1932 to M. G. Gregg; 1911644 of May 30, 1933 to F. C. Overbury; 1916885 of Jul. 4, 1933 to Lester Kirschbraun; 1969308 of Aug. 7, 1934 to Lester Kirschbraun; 2023540 of Dec. 10, 1935 to R. K. Painter, H. H. Morton and C. W. Hill; **Can. Pats.** 249764, 249765 and 249766 of May 19, 1925 to Flintkote Co.; 324466 of Jul. 26, 1932 to L. R. MacKenzie; 328665 of Dec. 20, 1932 to Flintkote Corp.; 352796 of Sep. 3, 1935 to Lancaster Processes, Inc.; **Brit. Pat.** 244135 of Jun. 10, 1924 to Lester Kirschbraun; 456955 of Mar. 18, 1935 to Compagnie Industrielle et Miniere du Nord et des Alpes.

4 [p. 505] **Ger. Pat.** Appl. N–32102 of May 25, 1931 to Erich Tobias.

5 [p. 506] **U. S. Pats.** 1330174 of Feb. 10, 1920 to J. A. DeCew; 1347734 of Jul. 27, 1920 to J. A. DeCew; 1432742 of Oct. 24, 1922 to F. J. Commin.

6 [p. 506] **Brit. Pat.** 379710 of Sep. 5, 1932 to "Emulgia" Vertriebsgesellschaft für Asphalt, Teer & Strassenbauprodukte, Clause & Co., Kommanditgesellschaft; **Ger. Pat.** 477760 of Jul. 9, 1925 to Paul Lechler; **French Pat.** 712875 of Mar. 5, 1931 to "Emulgia" Vertriebsgesellschaft für Asphalt, Teer & Strassenbauprodukte, Clause & Co., Kommanditgesellschaft.

7 [p. 507] "Asphalt Emulsion Paints," by J. M. Fain and Foster De Snell, *Paint, Oil & Chem. Rev.*, **98**, No. 13, 14 (1936); **U. S. Pat.** 1894617 of Jan. 17, 1933 to L. G. Gabriel; **Can. Pat.** 310873 of Apr. 28, 1931 to L. G. Gabriel; **Brit. Pat.** 320847 of May 22, 1928 to Colas Products Co., and L. G. Gabriel.

8 [p. 507] "Mechanical Dispersion by Means of the Colloidal Mill," by P. M. Travis, *J. Ind. Eng. Chem.*, **21**, 421 (1929); "The Preparation of Emulsions," by William Clayton, *J. S. C. I.*, **51**, 129 (1932); "Kolloidchemische Technologie der Asphalte und Teere," Handbuch Kolloidchemische Technologie," by R. E. Liesegang and Albrecht von Skopnik (1932); **U. S. Pat.** 1330174 of Feb. 10, 1920 to J. A. DeCew.

9 [p. 507] "Die Kolloidmühle und ihre Anwendungsmöglichkeiten," by Hermann Plauson, *Zeit. angew. Chem.*, **34**, 469 (1921); "A New Colloid Mill," by W. A. McLean, *J. Ind. Eng. Chem.*, **16**, 494 (1924); **U. S. Pats.** 1392849 of Oct. 4, 1921 to Hermann Plauson; 1436820 of Oct. 28, 1922 to Hermann Plauson; 1500845 of Jul 8, 1924 to Hermann Plauson; 1757065 of May 6, 1930 to H. E. Warsop and F. M. Gough; 1774205 of Aug. 26, 1930 to C. R. MacDonald; **Brit. Pats.** 185779 of Apr. 6, 1921 to Hermann Plauson; 196950 of Nov. 8, 1921 to J. A. Viellé; **Ger. Pats.** 385860 of Dec. 1, 1921 to Plauson's Forschungsinstitut, G.m.b.H. 390434 of Mar. 28, 1922 to Plauson's Forschungsinstitut, G.m.b.H.

10 [p. 507] **U. S. Pat.** 2036810 of Apr. 7, 1936 to G. C. Hurrell; **Can. Pat.** 316103 of Oct. 13, 1931 to Colas Roads, Inc.; **Ger. Pat.** 455593 of Mar. 5, 1926 to G. C. Hurrell.

11 [p. 507] **U. S. Pats.** 1807808 of Jun. 2, 1931 to H. L. Symons; 1851071 of Mar. 29, 1932 to P. M. Travis; 1988743 of Jan. 22, 1935 to L. R. MacKenzie.

12 [p. 507] **U. S. Pats.** 1848100 of Mar. 8, 1932 to H. P. Benner and J. C. Morrell; 1935884 of Nov. 21, 1933 to F. O. W. Loomis and D. M. Loomis; 1939391 of Dec. 12, 1933 to R. A. Curran.

13 [p. 508] **U. S. Pats.** 2009957 of Jul. 30, 1935 to J. F. Esch; 2015056 of Sep. 24, 1935 to J. L. Barnes; **Can. Pat.** 349975 of Apr. 30, 1935 to Texaco Development Corp.

14 [p. 508] **Can. Pat.** 251302 of Jun. 30, 1925 to Union Trust Co.; **Brit. Pat.** 238967 of May 31, 1924 to G. S. Hay.

15 [p. 508] **U. S. Pat.** 1384805 of Jul. 19, 1921 to D. J. McSwiney.

16 [p. 509] **Ger. Pats.** Appl. L-81753 of Aug. 3, 1932 to R. J. Lacau; Appl. L-83037 of Jan. 30, 1933 to R. J. Lacau.

17 [p. 509] "Teer und Bitumen in kolloidchemischer Betrachtungsweise," by H. Grohn, *Teer und Bitumen*, **27**, 345 and 365 (1929); "Bituminous Dispersions—Patent Review," by A. van der Wirth, *Asphalt Teer Strassenbautech.*, **34**, 1016 and 1037 (1934).

18 [p. 509] **U. S. Pats.** 1134573 of Apr. 6, 1915 to Herbert Abraham and H. W. Haines; 1382986 of Jun. 28, 1921 to L. M. Kreglius; 1384460 of Dec. 11, 1920 to C. B. Haley; 1607405 of Nov. 16, 1926 to H. S. Lukens; 1625304 of Apr. 19, 1927 to Dozier Finley; 1635095 of Apr. 24, 1924 to St. J. Robinson; 1759913 of May 27, 1930 to F. R. Moser; 1787418 of Dec. 30, 1930 to R. W. Lewis; 1869380, 1869381 and 1869382 of Aug. 2, 1932 to R. W. Lewis; **Can. Pat.** 268772 of Mar. 1, 1927 to F. R. Moser; **Brit. Pats.** 245418 of Jan. 2, 1925 to N. V. Bataafsche Petroleum Maatschappij; 273989 and 275928 of Mar. 18, 1927 to Rütgerswerke A.-G.; 346025 of Aug. 23, 1929 to D. J. Pickée; **Ger. Pats.** 40020 of Apr. 11, 1886 to Deutsche Asphalt A.-G.; 103733 of Jan. 13, 1898 to Hans Wunner; 275928 of Mar. 18, 1912 to Rütgerswerke A.-G.; 295064 of Mar. 17, 1915 to L. S. Van Westrum; 310893 of Oct. 20, 1915 to Rekord-Zement-Industrie, G.m.b.H.; 318699 of Aug. 25, 1917 to W. Klement; 335189 of Oct. 22, 1928 to G. Blasz & Sohn; 363246 of Jun. 26, 1918 to L. S. Van Westrum; 398793 of May 25, 1922 to Ernst Stern; 405930 of Nov. 22, 1922 to Ernst Stern; 422579 of Jul. 23, 1922 to Maschinenbauanstalt Humboldt; 433273 of Jan. 22, 1924 to N. V. de Bataafsche Petroleum Maatschappij; 484641 of Jan. 6, 1928 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger; 542148 of Sep. 15, 1927 to A. F. Malchow A.-G.

19 [p. 509] **U. S. Pat.** 903287 of Nov. 10, 1908 to H. V. D. Heide; **Brit. Pats.** of 1910 (Jul. 7), 16264 to T. D. Kelly; 185779 of Apr. 6, 1921 to Hermann Plauson; 433874 of Feb. 22, 1934 to I. G. Farbenindustrie A.-G.; **Ger. Pats.** 103287 of Nov. 10, 1908 to Hans Wunner; 103733 of Jan. 13, 1898 to Hans Wunner; 330650 of Jul. 6, 1918 to Harburger Chem. Werke Schön & Co. and W. Daitz; 398793 of May 25, 1922 to Ernst Stern; Appl. W-27653 of Apr. 29, 1907 to Hans Wunner (rejected); **Austrian Pat.** 116389 of Sep. 15, 1929 to Prax Chemische Versuchs- und Verwertungs G.m.b.H.

20 [p. 509] **U. S. Pat.** 1738776 of Dec. 10, 1929 to Lester Kirschbraun; **Brit. Pats.** 220393 of May 22, 1923 to F. E. Atherton; 454487 of Mar. 1, 1935 to A. S. for Kemisk Industri.

21 [p. 509] "Aqueous Mortars Containing Organic Binders," by E. Beyer, *Compt. rend.*, **198**, 2185 (1934); **Ger. Pats.** 542603 of Dec. 10, 1926 to Gesellschaft für Teerstrassen, m.b.H.; 561339 of May 10, 1931 to I. G. Farbenindustrie, A.-G.; **French Pat.** 751956 of Sep. 13, 1933 to S. A. des établissements Jean François.

22 [p. 509] "Water Mortars Containing Organic Binders," by E. Berger, *Compt. rend.*, **198**, 2185 (1934).

23 [p. 509] "Soluble Silicates in Industry," by J. G. Vail, Reinhold Publishing Corp., New York (1928); **U. S. Pats.** 21158 of Aug. 10, 1858 to Joseph Thompson; 32290 of May 14, 1861 to C. C. Hoff; 903287 of Nov. 10, 1908 to H. V. D. Heide; 980153 of Jan. 3, 1911 to Robert Hacking; 1171236 of Feb. 8, 1916 to E. A. Paterson; 1206056 of Nov. 28, 1916 to J. G. Vail; 1487471 of Mar. 18, 1924 to Rudolf Plönnis; 1668760 of May 8, 1928 to A. L. Clapp; 1738776 of Dec. 10, 1929 to Lester Kirschbraun; 1757102 of May 6, 1930 to P. M. Travis and A. L. Halvorsen; 1771744 of Jul. 29, 1930 to A. L. Clapp; 1989775 of Feb. 5, 1935 to P. R. Smith; 2051409 and 2051410 of Aug. 18, 1936 to J. A. Kenney; **Can. Pats.** 354903, 354904 and 354905 of Dec. 24, 1935 to Barrett Co.; **Brit. Pats.** 268411 of Dec. 3, 1925 to Frank Levy; 291502 of Feb. 28, 1927 to Bennett, Inc.; 323896 of Dec. 17, 1928 to Berry, Wiggins & Co., Ltd. and H. H. Holmes; 330374 of May 7, 1929 to Nils Bendixen and J. D. Morgan; 388093 of Sep. 9, 1931 to Robert Tagg; **Ger. Pats.** 316258 of Mar. 15, 1918 to W. Schmidt and E. Heuser; 336826 of May 29, 1918 to Dr. Plönnis & Co.; 349853 of Jul. 10, 1919 to Meilach Melamid and L. Grötzinger; 386168 of Apr. 29, 1921 to G. Borde; 399557 of Aug. 6, 1922 to Ernst Stern; 402173 of Jun. 16, 1923 to W. Kröger; 505106 of Apr. 6, 1927 to Michel Trux; Appl. C-38900 of Oct. 27, 1926 to Colas Kaltasphalt, G.m.b.H. (withdrawn); 606655 of Dec. 7, 1934 to Henkel & Cie., G.m.b.H.; **French Pats.** 594062 of Jan. 29, 1926 to F. G. Cros; 623725 of Oct. 27, 1926 to Asphalt Cold Mix, Ltd.; 623924 of Oct. 30, 1926 to Michel Trux.

24 [p. 509] **U. S. Pats,** 1707491 of Apr. 2, 1929 to R. P. Perry; 1747653 of Feb. 18, 1930 to Charlton Wilder; 2014700 of Sep. 17, 1935 to Joseph Salvi and G. S. Cunning; **Brit. Pat.** 262961 of Dec. 31, 1925 to Robert Illemann; **Ger. Pats.** 330650 of Jul. 6, 1918 to Harburger Chem. Werke Schön & Co. and W. Daitz; 575306 of Apr. 26, 1933 to G. A. Lasseur and T. V. Taralon.

25 [p. 509] **U. S. Pats.** 1000598 of Aug. 15, 1911 to J. B. Hall; 1049916 of Jan. 7, 1913 to Friedrich Raschig; 1134573 of Apr. 6, 1915 to Herbert Abraham and H. W. Haines; 1998769 and 1998955 of Sep. 19, 1916 to Clifford Richardson; 1240252 and 1240253 of Sep. 18, 1917 to M. A. Popkess; 1296083 of Mar. 4, 1919 to Robert Illemann; 1302810 of May 6, 1919 to Lester Kirschbraun; 1305645 of Jun. 3, 1919 to Samuel Cabot; 1320709 of Nov. 4, 1919 to M. A. Popkess; 1327726 of Jan. 13, 1920 to S. R. Murray; 1330443 of Feb. 10, 1920 to G. C. Glynn; 1398201 of Nov. 22, 1920 to C. S. Reeve; 1408224 of Jul. 23, 1921 to C. S. Reeve; 1417835 and 1417837 of May 30, 1922 to Lester Kirschbraun; 1461445 of Jul. 10, 1923 to Lester Kirschbraun; 1469563 of Oct. 2, 1923 to Lester Kirschbraun; 1479042 of Jan. 1, 1924 to Lester Kirschbraun; 1495260 of May 27, 1924 to Prevost Hubbard, C. S. Reeve and R. H. Lewis; 1506371 of Aug. 26, 1924 to Lester Kirschbraun; 1517075 of Nov. 25, 1924 to Lester Kirschbraun; 1567061 of Dec. 29, 1925 to Lester Kirschbraun; Reissue 16328 of Apr. 20, 1926 to Lester Kirschbraun; 1609308 of Dec. 7, 1926 to W. B. Pratt; 1615303 of Jan. 25, 1927 to Lester Kirschbraun; 1616904 of Feb. 8, 1927 to Lester Kirschbraun; 1620899 and 1620900 of Mar. 15, 1927 to Lester Kirschbraun; 1663652 af Mar. 27, 1928 to H. R. Gundlach; 1670844 of May 22, 1928 to M. R. Cone; 1679475 of Aug. 7, 1928 to Lester Kirschbraun; 1691765 of Nov. 13, 1928 to Lester Kirschbraun; 1691766 and 1691767 of Nov. 13, 1928 to Lester Kirschbraun and H. L. Levin; 1691768 of Nov. 13, 1928 to Lester Kirscbhraun; 1699573 of Jan. 22,

1929 to L. C. Jones, C. C. Loomis and H. W. Banks; 1707809 of Apr. 2, 1929 to Lester Kirschbraun; 1722433 of Jul. 30, 1929 to Lester Kirschbraun; 1734437 of Nov. 5, 1929 to Lester Kirschbraun; 1751690 of Mar. 25, 1930 to C. R. Faben; 1752449 of Apr. 1, 1930 to J. S. Miller, Jr.; 1824428 of Sep. 22, 1931 to A. Fisher; 1882834 of Oct. 18, 1932 to L. A. Hall and E. E. Mortell; 1960112 of May 22, 1934 to Lester Kirschbraun and H. L. Levin; 1980192 of Nov. 13, 1934 to J. M. Fain; **Can. Pats.** 132373 of Apr. 11, 1911 to Friedrich Raschig; 207413 of Jan. 11, 1921 to Lester Kirschbraun; 245710, 245712 and 245715 of Dec. 30, 1924 to Lester Kirschbraun; 245725 of Dec. 30, 1924 to G. J. Manson; 248144 and 248220 of Mar. 31, 1925 to Lester Kirschbraun; 249764, 249765 and 249766 of May 19, 1925 to Lester Kirschbraun; 256081 of Dec. 8, 1925 to Lester Kirschbraun; 276936 of Jan. 10, 1928 to Lester Kirschbraun; 279241 of Apr. 10, 1928 to Lester Kirschbraun; 280289 of May 22, 1928 to Lester Kirschbraun; 309053 and 309054 of Mar. 3, 1931 to Lester Kirschbraun and H. L. Levin; 310872 of Apr. 28, 1931 to Lester Kirschbraun; **Brit. Pats.** of 1908 (Jul. 16), 15100 to Julius Kathe; of 1910 (May 4), 15711 to Friedrich Raschig; 117785 of Mar. 6, 1918 to Robert Illemann and J. A. Montgomerie; 206888 of Jul. 11, 1922 to T. Murphy; 243976 of Feb. 5, 1925 to Wales Dove Bitumastic, Ltd. and R. Shaw; 291276 of Oct. 20, 1927 to Friedrich Raschig; 315495 of Apr. 25, 1928 to G. C. Hurrell; 321721 of May 21, 1928 to F. B. Dehn; 334100 of Oct. 9, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 343427 of Oct. 9, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 351073 of May 9, 1930 to C. M. C. Hughes and C. W. Fulton; 443666 of Apr. 1, 1935 to Paul Lechler; **Ger. Pats.** 68532 of Jul. 1, 1891 to Grünzweig & Hartmann; 211877 of Sep. 5, 1906 to Julius Kathe; 216212 of Jun. 25, 1907 to Freidrich Raschig; 244307 of May 5, 1910 to Friedrich Raschig; 296271 of Aug. 3, 1916 to Carl Roth; 298708 of Oct. 8, 1916 to Carl Roth; 495232 of Jun. 2, 1927 to Friedrich Raschig, G.m.b.H.; 582794 of Aug. 23, 1933 to Flintkote Co.; Appl. E–18313 of Aug. 8, 1912 to Ludwig Esselborn (rejected); **French Pats.** 592956 of Jul. 11, 1922 to F. Simon; 637796 of Jul. 19, 1927 to Soc. d'Etudes Chimiques; 655355 of May 29, 1928 to Flintkote Co.; 683991 of Oct. 28, 1929 to N.V. de Bataafsche Petroleum Maatschappij; **Austrian Pat.** 116390 of Sep. 15, 1929 to Prax Chemische Versuchs- und Verwertungs-G.m.b.H.; **Danish Pat.** 50735 of Oct. 7, 1935 to Aktieselskabet for Kemisk Industri; "Water-soluble Coal Tar," by Friedrich Raschig, *Chem. Rev. Fett- und Harz-Ind.*, 17, 169 (1910); "Water-soluble Coal Tar for Treating Roads," by Friedrich Raschig, *Z. angew. Chem.*, 23, 973 (1910); also *J. Soc. Chem. Ind.*, 27, 758 (1910); "A Study of Water-miscible Mineral-oil Preparations," by Ralph Hart, *J. Ind. Eng. Chem.*, 21, 85 (1929).

26 [p. 509] "Bentonite Helps Solve Problem in Grinding Asphalt," by H. S. Spence, *Chem. Met. Eng.*, 30, 986 (1924); "Bentonite, Its Properties, Mining, Preparation and Utilization," by C. W. Davis and H. C. Vacher, Tech. Paper 438, Bureau of Mines, Washington, D. C. (1928).

27 [p. 509] **U. S. Pat.** 2037669 of Apr. 14, 1936 to R. R. Thurston; **Ger. Pat.** 530420 of Nov. 15, 1927 to W. H. Schmitz.

28 [p. 509] **U. S. Pats.** 1711727 of May 7, 1929 to C. N. Forrest; 1783365 of Dec. 2, 1930 to R. W. Lewis; 1788748 of Jan. 13, 1931 to A. E. Schutte; **Ger. Pat.** 118992 of Jun. 12, 1900 to Lazar Rosenfeld.

29 [p. 509] **U. S. Pats.** 956009 of Apr. 26, 1910 to L. S. Van Westrum; Reissue 15401 of Jul. 4, 1922 to L. S. Van Westrum.

30 [p. 509] **U. S. Pat.** 984518 of Feb. 14, 1911 to Roy Anderson and W. E. Moltz.

31 [p. 509] **U. S. Pats.** 1714982 of May 28, 1929 to A. L. Halvorsen and P. M. Travis; 1757083 of May 6, 1930 to A. L. Halvorsen and P. M. Travis; 1757102 and 1757103 of May 6, 1930 to P. M. Travis and A. L. Halvorsen; 1926246 and 1926247 of Sep. 12, 1933 to P. R. Smith; **Can. Pat.** 293162 of Sep. 17, 1929 to Emulsion Process Corporation; **Brit. Pats.** 300414 and 300415 of Dec. 19, 1927 to J. A. Montgomerie; 307288 of May 3, 1928 to A. L. Halvorsen and P. M. Travis; 353702 of Aug. 16, 1929 to Metallgesellschaft, A.-G.; 427720 of Jun. 26, 1933 to F. B. Dehn; **Ger. Pat.** 521593 of Jun. 9, 1926 to Heinrich Kretzer.

32 [p. 509] **U. S. Pat.** 1860248 of May 24, 1932 to J. Hyman and A. F. Schlandt; **Brit. Pat.** 319663 of Sep. 25, 1928 to C. G. J. Lefebvre and E. E. F. Berger.

33 [p. 509] "Emulsions with Finely Divided Solids," by T. R. Briggs, *J. Ind. Eng. Chem.*, 13, 1008 (1921).

34 [p. 509] **U. S. Pats.** 73531 of Jan. 21, 1868 to J. A. Jones; 118296 of Aug. 22, 1871 to W. M. Stuart; 461467 of Oct. 20, 1891 to M. O. Farrar and C. C. Howe; 998691 of Jul. 25, 1911 to H. R. Kasson and S. S. Saxton; 1127831 of Feb. 9, 1915 to L. S. Van Westrum; 1643675 of Sep. 27, 1927 to J. A. Montgomerie; 1723844 of Aug. 6, 1929 to W. P. Davey; 1737491 of Nov. 26, 1929 to C. A. Braun; 1915062 of Jun. 20, 1933 to Hyman Limburg; 1973692 of Sep. 18, 1934 to H. L. Becher; 1981522 of Nov. 20, 1934 to O. F. Neitzke; 1994542 of Mar. 19, 1935 to P. R. Smith and O. R. Douthett; **Can. Pats.** 251421 of Jul. 7, 1925 to J. A. Montgomerie; 264634 of Sep. 28, 1926 to Asphalt Cold Mix, Ltd.; 286843 of Jan. 29, 1929 to Crown Bitumens Corporation; **Brit. Pats.** 226032 of Dec. 8, 1923 to J. A. Montgomerie; 288821 of Apr. 23, 1927 to J. A. Montgomerie; 298842 of Jan. 26, 1928 to Chem. Fabrik Buckau; 332591 of Apr. 22, 1929 to Richard Lichtenstern; 392365 of Jun. 9, 1932 to Paul Lechler; 421269 of Apr. 6, 1933 to Eugéne Rouault; **Ger. Pats.** 52129 of May 8, 1889 to W. Dammann; 143620 of Apr. 2, 1902 to L. S. Van Westrum; 251710 of Feb. 9, 1912 to Carl Roth; 258608 of Mar. 12, 1912 to Rütgerswerke A.-G.; 316258 of Mar. 15, 1918 to W. Schmidt and E. Heuser; 335189 of Oct. 22, 1918 to G. Blasz & Sohn; 335996 of Oct. 3, 1917 to H. Th. Böhme A.-G., Chem. Fabrik; 349853 of Jul. 10, 1919 to Meilach Melamid and L. Grötzinger; 407106 of Feb. 15, 1924 to Norddeutsche Portland-Cement-Fabrik Misburg and Willy Renner; 433273 of Jan. 22, 1924 to N. V. de Bataafsche Petroleum Maatschappij; 499713 of Jan. 22, 1925 to Bitumuls Kaltasphalt A.-G.; 542148 of Sep. 15, 1927 to A. F. Malchow A.-G.; 587829 of Nov. 9, 1933 to Société Chimique; 589960 of Mar. 12, 1931 to "Emulgia" Vertriebsgesellschaft für Asphalt-Teer- und Strassenbauprodukte, Clause & Co. Kommanditgesellschaft; 603801 of Jan. 8, 1929 to Erwin Bergel; Appl. M–120786 of Feb. 19, 1932 to Main- Gaswerke A.-G.; **French Pats.** 588886 of Nov. 14, 1924 to J. A. Montgomerie; 351170 (addition to 588886) of Mar. 30, 1928 to International Colfix, Ltd.; 603136 of Sep. 14, 1925 to C. A. Braun; 683116 of Oct. 11, 1929 to T. W. Barber; **Austrian Pats.** 89638 of May 4, 1920 to Meilach Melamid and L. Grötzinger; 134983 of Oct. 25, 1933 to Erwin Bergel; **Swiss Pats.** 118707 of Sep. 17, 1925 to G. S. Hay & Co., Ltd.; 122055 of Feb. 12, 1926 to Mineral A.-G.; 123737 of Mar. 1, 1926 to N. V. de Bataafsche Petroleum Maatschappij.

35 [p. 509] **U. S. Pats.** 1803488 of May 5, 1931 to E. S. Ross; 2013972 of Sep. 10, 1935 to S. S. Sadtler and W. H. Field.

36 [p. 509] **Brit. Pat.** 233430 of Feb. 8, 1924 to H. A. Mackay.

37 [p. 509] **Ger. Pat.** 589015 of Dec. 1, 1933 to Carl Stiepel.

38 [p. 509] **Ger. Pat.** 521593 of Jun. 9, 1926 to Heinrich Kretzer.

39 [p. 509] **U. S. Pat.** 1914100 of Jun. 13, 1933 to Harry Bennett; **Can. Pats.** 322765 of May 31, 1932 to Harry Bennett; 336186 of Oct. 3, 1933 to Harry Bennett.

40 [p. 509] **Brit. Pats.** 312372 of Nov. 18, 1927 to G. Plauson; 438162 of Nov. 20, 1934 to J. R. Geigy A.-G.; **Ger. Pats.** 301927 of Dec. 19, 1916 to Georg Muth; 302632 of Dec. 5, 1916 to Georg Muth; 305271 of Jun. 21, 1917 to Georg Muth; 316345 of Jun. 6, 1916 to Georg Muth; 316617 of Apr. 23, 1919 to Georg Muth.

41 [p. 510] "Utilization of Resinous Products for Road Surfacing," by G. Brus and M. Barraud, *Bull. inst. pin.*, 11 (1935); **U. S. Pats.** 683735 of Oct. 1, 1901 to Sam Dyson and John Gaskell; 713190 of Nov. 11, 1902 to C. L. V. Zimmer; 790831 of May 23, 1905 to H. V. Dunham; 834830 of Oct. 30, 1906 to Karl Mann; 992313 of May 16, 1911 to L. S. Van Westrum; 998691 of Jul. 25, 1911 to H. R. Kasson and S. S. Saxton; 1014103 of Jan. 9, 1912 to Reinhold Wallbaum; 1077081 of Oct. 28, 1913 to F. O. Kaempf; 1127831 of Feb. 9, 1915 to L. S. Van Westrum; 1258103 of Mar. 5, 1918 to W. M. Fraser; 1259223 of Mar. 12, 1918 to W. M. Fraser; 1266335 of May 14, 1918 to Buel Smith; 1266955 and 1266956 of May 21, 1918 to Bruno Kniffler; 1384805 of Mar. 5, 1920 to F. R. Mullin; 1432742 of Oct. 24, 1922 to F. J. Commin; 1472716 of Oct. 30, 1923 to W. P. Davey; 1542626 of Jun. 16, 1925 to H. A. Mackay; 1565125 of Dec. 8, 1925 to L. S. Van Westrum; 1609308 of Dec. 7, 1926 to W. B. Pratt; 1640544 of Aug. 30, 1927 to W. T. Headley; 1665015 of Apr. 3, 1928 to Robert Mezger; 1665881 of Apr. 10, 1928 to Eugen Hutzenlaub; 1699536 of Jan. 22, 1929 to L. C. Jones, C. C. Loomis and H. W. Banks; 1706590 of Mar. 26, 1929 to Hermann Plauson; 1752449 of Apr. 1, 1930 to J. S. Miller, Jr.; 1861398 of May 31, 1932 to R. Lant; 1861826 of Jun. 7, 1932 to L. G. Thompson; 1931072 of Oct. 17, 1933 to D. McK. Hepburn; 1940431 and 1940432 of

Dec. 19, 1933 to O. F. Neitzke; 1957031 of May 1, 1934 to D. N. Meyers; 1973599 of Sep. 11, 1934 to C. M. Baskin; 1988336 of Jan. 15, 1935 to J. C. Roediger; 2003860 of Jun. 4, 1935 to K. E. McConnaughey; 2027404 of Jan. 14, 1936 to J. B. Small; 2053099 of Sep. 1, 1936 to Jack Miscall; **Can. Pats.** 229000 of Feb. 27, 1923 to F. J. Commin; 236318 of Dec. 11, 1923 to H. A. Mackay; 276480 of Dec. 20, 1927 to Mineral A.-G.; 279232 of Apr. 10, 1928 to W. T. Headley; 299042 of Apr. 8, 1930 to J. A. Montgomerie; 300450 of May 20, 1930 to F. W. Atack; 323861 of Jul. 5, 1932 to Bennett, Inc.; 326277 of Sep. 27, 1932 to C. D. Cory; 341465 of May 8, 1934 to Bennett, Inc.; 354233 of Nov. 19, 1935 to Barrett Co.; 365239 of Apr. 6, 1937 to Flintkote Co.; **Brit. Pats.** of 1905 (Jun. 2), 11191 to E. Braschler-Kürtz; 139491 and 139492 of Feb. 25, 1920 to W. M. Fraser; 155398 of Sep. 30, 1919 to E. Schou; 187779 of Aug. 30, 1921 to John Hines; 202021, 202030, 202031, 202035 of Sep. 5, 1922 to H. A. Mackay; 202231 and 202235 of May 9, 1922 to H. A. Mackay; 202393 of May 22, 1924 to F. E. Atherton; 232683 of Jan. 23, 1924 to H. A. Mackay; 233371 of Nov. 8, 1923 to L. S. Van Westrum; 233430 of Feb. 8, 1924 to H. A. Mackay; 238967 of May 31, 1924 to G. S. Hay; 251148 of Jul. 24, 1925 to B. C. Sellers; 251323 of Jan. 1, 1925 to G. S. Hay; 252260 of Feb. 24, 1925 to G. S. Hay; 252449 of Nov. 27, 1924 to W. E. Billinghame; 258870 of Sep. 20, 1926 to E. B. Hack; 263307 of Jan. 5, 1926 to C. H. Thompson and W. J. McGivern; 264496 of Jan. 11, 1927 to Soc. anom. La Trinidad; 276543 of Dec. 3, 1926 to Mineral A.-G.; 279026 of Jun. 13, 1927 to Soc. anom. La Trinidad; 292251 of Mar. 17, 1927 to Asphalt Cold Mix, Ltd.; 305716 of Nov. 11, 1927 to J. A. Montgomerie; 308051 of Feb. 16, 1928 to J. A. Montgomerie; 315057 of May 23, 1928 to C. H. Thompson and W. J. McGivern; 319101 of Jul. 25, 1928 to Hermann Plauson; 319663 of Sep. 18, 1929 to C. G. J. Lefebvre and E. E. F. Berger; 320357 of Jul. 9, 1928 to Josef Klein; 333152 and 333153 of Feb. 4, 1929 to C. G. Fox; 333496 of Feb. 2, 1919 to Paul Lechler; 344562 of Dec. 18, 1929 to J. Leben and Ormul Products, Ltd.; 379710 of Mar. 3, 1932 to A. L. Mond; 382432 of Nov. 2, 1931 to Hanseatische Mühlenwerke, A.-G.; 387825 of Jan. 18, 1932 to H. A. Gill; 441782 of Aug. 2, 1934 to Colas Products, Ltd., L. G. Gabriel and J. F. T. Blott; **Ger. Pats.** 52129 of May 8, 1889 to W. Dammann; 122451 of Jun. 6, 1899 to F. Boleg; 129480 of May 1, 1900 to F. Boleg; 170133 of Jun. 3, 1904 to Karl Mann; 248084 of Dec. 5, 1909 to Reinhold Wallbaum; 248793 of Mar. 17, 1910 to Reinhold Wallbaum; 295893 of Jan. 19, 1910 to W. H. Elmenhorst; 308442 of Jan. 25, 1917 to St. Rochus, G.m.b.H.; 321113 of Jul. 3, 1917 to E. Stephani; 342639 of Dec. 24, 1920 to F. J. Commin; 343575 of Feb. 28, 1918 to K. Winkler & Co.; 352357 of Sep. 1, 1918 to Carl Suchy and Rudolf Luszak; 357378 of Feb. 8, 1921 to Chemische Werkstätten; 363246 of Jun. 26, 1918 to L. S. Van Westrum; 368233 of Jun. 11, 1921 to R. Houben; 378385 of Aug. 16, 1922 to Ernst Stern; 392337 of Feb. 19, 1921 to Plauson's Forschungsinstitut, G.m.b.H.; 398879 of Oct. 3, 1922 to Chemische Werkstätten; 407199 of Apr. 26, 1924 to Paul Lechler; 459655 of Feb. 1, 1925 to L. S. Van Westrum; 470306 of May 5, 1923 to Asphalt Cold Mix, Ltd.; 498425 of Feb. 15, 1927 to K. Meisenhelder; 506135 of Dec. 31, 1927 to J. A. Montgomerie; 541439 of Jun. 19, 1931 to Michael Groskopf; 542605 of Dec. 10, 1926 to Gesellschaft für Teerstrassenbau, m.b.H.; 549263 of Mar. 5, 1929 to Gesellschaft für Teerverwertung, m.b.H. and Fritz Kraft; 553245 of Feb. 3, 1929 to Paul Lechler; 554995 of Nov. 30, 1928 to Berthold Redlich; 556250 of May 5, 1928 to Deutsche Erdöl, A.-G.; 557154 of Nov. 16, 1930 to Hanseatische Mühlenwerke, A.-G.; 557288 of Aug. 17, 1930 to Gesellschaft für Teerverwertung, m.b.H., Fritz Kraft and Theodor Heydecke; 566670 of Nov. 15, 1929 to Chem. Fabrik Flörsheim, Dr. H. Noerdlinger, A.-G.; 571488 of Feb. 8, 1931 to Main- Gaswerke A.-G., Gas Passage and H. W. Hoelzer; 574528 of Feb. 20, 1932 to Main- Gaswerke A.-G. and H. W. Hoelzer; 575104 of Dec. 21, 1926 to Heinrich Kretzer; 584540 of Dec. 24, 1927 to Wilhelm Ackermann; 584695 of Oct. 14, 1928 to Heinrich Kretzer; 593184 of Mar. 12, 1929 to Gesellschaft für Teerstrassenbau, m.b.H.; 594189 of Apr. 16, 1933 to Hanseatische Mühlenwerke, A.-G.; 602165 of Nov. 21, 1933 to J. R. Geigy, A.-G.; Appl. S–77952 of Jan. 12, 1927 to S. A. La Trinidad (withdrawn); Appl. T–35775 of Oct. 1, 1928 to Trinidad deutsche Oel- und Asphalt, A.-G. (withdrawn); Appl. C–45004 of Jun. 24, 1931 to Colas Flintkote, Ltd.; Appl. J–43070 of Nov. 11, 1931 to I. G. Farbenindustrie, A.-G.; Appl. G–88052 of May 11, 1934 to J. R. Geigy, A.-G.; 631781 of Apr. 6, 1933 to Eugène Rouault; 632300 of Jan. 10, 1933 to R. O. Bratke; Appl. D–69929 of Mar. 12, 1935 to Erwin Dornig; **French Pats.**

358792 of Oct. 24, 1905 to B. Loyeau; 564943 of Apr. 10, 1923 to Asphalt Cold Mix, Ltd.; 36752 (addition to 564943) of Mar. 23, 1929 to Asphalt Cold Mix, Ltd.; 577488 of Aug. 3, 1926 to Thompson-Houston Cie.; 594062 of Jan. 29, 1926 to F. G. Cros; 597925 of Aug. 14, 1924 to Soc. de Recherches; 603136 of Sep. 14, 1925 to C. A. Braun; 608123 of Dec. 21, 1925 to Soc. Rol. Lister et Cie.; 609140 of Jan. 12, 1926 to Soc. anom. La Trinidad; 609966 of Jan. 25, 1926 to Soc. anom. La Trinidad; 611479 of Feb. 19, 1926 to Union française de Crédit; 613084 of Jul. 9, 1925 to Georges Baume, Pierre Chambige and de Boutier; 623924 of Oct. 30, 1926 to Michel Trux; 630168 of Mar. 4, 1927 to H. Basset and V. Szidon; 636207 of Oct. 15, 1926 to Soc. anom. La Trinidad; 684048 of Oct. 29, 1929 to Agasote Millboard Co.; 744008 of Apr. 11, 1933 to Bitumen Investments, Inc.; 752178 of Mar. 9, 1933 to Fernando Elosegui; 771284 of Oct. 4, 1934 to Hanseatische Mühlenwerke, A.-G.; 775229 of Dec. 21, 1934 to Alfred Tabary; 781161 of May 10, 1935 to Société Paix & Cie.; 781331 of May 13, 1935 to J. R. Geigy, S. A.; 792874 of Jan. 11, 1936 to N. V. de Bataafsche Petroleum Maatschappij; **Austrian Pat.** 137894 of Jun. 11, 1934 to Richard Lichtenstern; **Swiss Pats.** 116806 of May 6, 1925 to Asphalt Cold Mix, Ltd.; 120508 of Feb. 1, 1926 to Asphalt Cold Mix, Ltd.; 122056 of Feb. 12, 1926 to Mineral A.-G.; 164109 of Dec. 1, 1933 to Hermann Plauson; **Swedish Pat.** 57425 of Apr. 19, 1923 to Asphalt Cold Mix, Ltd.; **Australian Pat.** 23721 of Mar. 26, 1936 to Asphalt Cold Mix, Ltd.

42 [p. 510] "New Commercial Emulsifying Agents," by Harry Bennett, *J. Ind. Eng. Chem.*, **22**, 1255 (1930); **Brit. Pat.** 437674 of Nov. 4, 1935 to E. I. Dupont de Nemours & Co.; **French Pat.** 717390 of May 20, 1931 to Frédéric Steinfels (S.A.).

43 [p. 510] **U. S. Pat.** 1988879 of Jan. 22, 1935 to H. M. Steininger; **Brit. Pat.** 381286 of Aug. 16, 1931 to N. V. Tot Voortzetting der Zaken van Pieter Schoen & Zoon.

44 [p. 510] **Ger. Pats.** 545763 of Mar. 10, 1929 to Th. Goldschmidt, A.-G.; 551403 of Aug. 28, 1927 to Th. Goldschmidt, A.-G.; 582106 of Aug. 9, 1933 to Th. Goldschmidt, A.-G.; 590165 of Feb. 15, 1928 to Th. Goldschmidt, A.-G.

45 [p. 510] **U. S. Pat.** 1938804 of Dec. 12, 1933 to Mendel Burak.

46 [p. 510] **French Pat.** 745113 of May 5, 1933 to I. G. Farbenindustrie, A.-G.

47 [p. 510] **U. S. Pats.** 931520 of Aug. 17, 1909 to Julius Stockhausen; 998691 of Jul. 25, 1911 to H. R. Kasson and S. S. Saxton; 1010210 of Nov. 28, 1911 to K. L. V. Zimmer; 1237852 of Aug. 21, 1917 to C. L. V. Zimmer; 1319918 of Oct. 28, 1919 to C. L. V. Zimmer; 1391710 of Sep. 27, 1921 to C. L. V. Zimmer; 1875001 of Aug. 30, 1932 to A. B. Hoel; 1888295 of Nov. 22, 1932 to P. R. Smith; **Can. Pat.** 254598 of Oct. 13, 1925 to Asphalt Cold Mix, Ltd.; **Brit. Pats.** of 1915 (Aug. 5), 11331 to C. L. V. Zimmer; 236641 of Apr. 8, 1924 to H. A. Mackay; 252258 of Feb. 23, 1925 to H. W. Hutton and C. W. Fulton; 258870 of Sep. 20, 1926 to E. B. Hack; 271777 of Feb. 24, 1926 to H. E. Potts; 331153 of Feb. 4, 1929 to C. F. Fox; 340294 of Jul. 27, 1929 to J. Y. Johnson; 365229 of Jan. 5, 1931 to Imperial Chemical Industries, Ltd.; 441601 of Jul. 20, 1934 to Deutsche Hydrierwerke A.-G.; **Ger. Pat.** 527935 of Sep. 13, 1929 to Westfälische Mineralöl- und Asphaltwerke, W. H. Schmitz Komm.-Ges.; **French Pats.** 595151 of Mar. 14, 1925 to Asphalt Cold Mix, Ltd.; 598131 of May 4, 1925 to G. F. Jardin; 611479 of Feb. 19, 1926 to Union française de Credit; **Swiss Pats.** 106549 of Oct. 12, 1923 to R. Maag; 119219 of May 27, 1925 to H. Matti; **Swedish Pat.** 64738 of Mar. 5, 1925 to Asphalt Cold Mix, Ltd.

48 [p. 510] **Brit. Pat.** 389810 of Jun. 18, 1931 to Colas Products, Ltd.

49 [p. 510] "Dust Preventatives and Road Binders," by Prévost Hubbard, pp. 107–8 (1910); **U. S. Pats.** 865578 of Sep. 10, 1907 to Carleton Ellis; 1107020 of Aug. 11, 1914 to Ludwig Berend; 1236875 of Aug. 14, 1917 to J. S. Robeson; 1431165 of Oct. 10, 1922 to H. S. Levenson; 1444844 of Feb. 13, 1923 to K. P. McElroy; 1450685 of Apr. 3, 1923 to Lester Kirschbraun; Reissue 15944 of Nov. 11, 1924 to Lester Kirschbraun; 1662299 of Dec. 19, 1927 to M. F. Coughlin; 1840157 of Jan. 5, 1932 to C. F. Cross; 1842893 of Jan. 26, 1932 to W. E. B. Baker and Harry Weaver; 1936250 of Nov. 21, 1933 to Hyman Limberg; 2040208 of May 12, 1936 to R. J. Lacau; **Can. Pat.** 247469 of Mar. 10, 1925 to Lester Kirschbraun; **Brit. Pats.** of 1912 (Jun. 10), 13586 to Kurt Albert and Ludwig Berend; of 1915 (Nov. 19), 16315 to Ludwig Berend; 263307 of Jan. 5, 1926 to C. H. Thompson and W. J. McGivern; 263520 of Sep. 21, 1925 to C. F. Cross and Alf Engelstad; 280930 of Nov. 16, 1927 to Georges

Baume; 300821 of Dec. 24, 1927 to J. A. Montgomerie; 307000 of Dec. 1, 1927 to J. R. Geigy, A.G.; **Ger. Pats.** 248793 of Mar. 17, 1910 to Reinhold Wallbaum; 250275 of Jun. 13, 1911 to Kurt Albert and Ludwig Berend; 321213 of Jun. 21, 1918 to Deutsche Succol G.m.b.H.; 342639 of Dec. 24, 1920 to F. J. Commin; 539165 of Apr. 5, 1928 to Chemische Fabriken, Kurt Albert, G.m.b.H.; 582997 of Aug. 4, 1932 to R. J. Lacau; 583196 of Jul. 22, 1932 to Romag A.-G. für Rohmaterialien; Appl. C–42744 of Mar. 8, 1929 to Chemische Fabriken, Kurt Albert, G.m.b.H.; 584936 of Jan. 3, 1933 to R. J. Lacau; **French Pat.** 628302 of Nov. 26, 1916 to G. Murphy; **Austrian Pat.** Appl. N–7635 of Sep. 10, 1909 to Romauld Nowiciki; 72451 of Sep. 25, 1916 to Kurt Albert and Ludwig Berend; **Danish Pat.** 23419 of Aug. 22, 1918 to O. Lührs.

50 [p. 510] **U. S. Pat.** 1167373 of Jan. 11, 1916 to Ludwig Berend.

51 [p. 510] **U. S. Pat.** 1895775 of Jan. 31, 1933 to P. R. Smith.

52 [p. 510] **Ger. Pat.** 590723 of Dec. 21, 1933 to Deutsche Vialit, G.m.b.H.

53 [p. 510] **Brit. Pats.** 268387 of Mar. 28, 1927 to I. G. Farbenindustrie, A.-G.; 353926 of Aug. 29, 1930 to I. G. Farbenindustrie, A.-G.; **Ger. Pats.** 431249 of Dec. 24, 1924 to I. G. Farbenindustrie, A.-G.; 543326 of Dec. 12, 1929 to I. G. Farbenindustrie, A.-G.

54 [p. 510] "Neuere Sulphonierungsverfahren zur Herstellung von Dispergier-, Netz- und Waschmitteln," by A. van der Werth and F. Mueller, 2nd Edition, Allgemeiner Industrie-Verlag, G.m.b.H., Berlin (1936); "Die Naphthensäuren," by J. Budowski, Verlag von Julius Springer, Berlin (1922); "Naphthensäuren und Naphthensulfosäuren" (Narchtrag 1927–1933, zu "Chemie, Technologie und Analyze der Naphthensäuren"), by M. Naphtali, Wissenschaftliche Verlagsgesellschaft, m.b.H., Stuttgart (1934); **U. S. Pats.** 1211721 of Jan. 9, 1917 to Ludwig Landsberg; 1373661 of Apr. 15, 1921 to E. M. Johansen; 1464928 of Aug. 14, 1923 to C. N. Forrest; 1695197 of Dec. 11, 1928 to D. R. Merrill; 1739686 of Dec. 17, 1929 to D. R. Merrill; 1750198 of Mar. 11, 1930 to Walther Schrauth; 1773123 of Aug. 19, 1930 to F. W. Sullivan, Jr.; 1817599 of Aug. 4, 1931 to E. W. Adams and F. W. Kittrell; 1830969 of Nov. 10, 1931 to F. W. Sullivan, Jr. and E. W. Adams; 1843420 of Feb. 2, 1932 to Fritz Günther, Fritz Teller, Carl Immerheiser and Bodo Zschimmer; 1871939 and 1871940 of Aug. 16, 1932 to E. W. Adams; 1905630 of Apr. 25, 1933 to O. R. Douthett; 1907920 of May 9, 1933 to R. E. Wilkin; 1920787 of Aug. 1, 1933 to E. J. Hammers and Egon Eichwald; 1921158 of Aug. 8, 1933 to E. T. Hessle; 1931880 of Oct. 24, 1933 to H. F. Angstadt; 1940807 of Dec. 26, 1933 to Leo Liberthson; 1969491 of Aug. 7, 1934 to E. W. Adams; 2013619 of Sep. 3, 1936 to H. F. Angstadt; **Can. Pats.** 253013 of Aug. 25, 1925 to Asphalt Cold Mix, Ltd.; 291110 of Jul. 9, 1929 to Hyman Limberg; 292556 of Aug. 27, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 305502 of Nov. 4, 1930 to E. R. De Ong; 317471 of Nov. 24, 1931 to Rütgerswerke, A.-G.; 330017 of Feb. 7, 1933 to Flintkote Corp.; **Brit. Pats.** 233784 of Feb. 14, 1924 to H. A. Mackay; 287514 of Mar. 23, 1927 to A. W. Burwell; 291393 of May 7, 1928 to N. V. de Bataafsche Petroleum Maatschappij and H. Limberg; 300574 of Nov. 15, 1927 to N. V. de Bataafsche Petroleum Maatschappij; 302346 of Nov. 13, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 320357 of Jul. 9, 1928 to Josef Klein; 360002 of Jul. 21, 1930 to I. G. Farbenindustrie, A.-G.; **Ger. Pats.** 248084 and 248793 of Mar. 17, 1910 to Reinhold Wallbaum; 267126 of May 1, 1912 to Ludwig Landsberg; 378385 of Aug. 16, 1922 to Ernst Stern; 385860 of Dec. 1, 1921 to Plauson's Forschungsinstitut, G.m.b.H.; 390434 of Mar. 28, 1922 to Plauson's Forschungsinstitut, G.m.b.H.; 394107 of Nov. 25, 1921 to Sudfeldt & Co.; 438180 of Sep. 9, 1922 to I. G. Farbenindustrie, A.-G.; 445099 of Oct. 7, 1922 to I. G. Farbenindustrie, A.-G.; 466567 of Nov. 18, 1925 to Hermann Plauson; 511456 of Oct. 8, 1926 to Verkaufsvereinigung für Teererzeugnisse, G.m.b.H.; 518394 of Aug. 2, 1926 to Verkaufsvereinigung für Teererzeugnisse, G.m.b.H.; 530420 of Nov. 15, 1927 to W. H. Schmitz; 536912 of May 3, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 542148 of Sep. 15, 1927 to A. F. Malchow A.-G.; 542634 of Oct. 7, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 551402 of Sep. 19, 1925 to I. G. Farbenindustrie A.-G.; 566797 of Feb. 20, 1930 to I. G. Farbenindustrie A.-G.; 574536 of Apr. 18, 1933 to Kurt Linder and Johannes Zickermann; 588139 of Apr. 20, 1930 to I. G. Farbenindustrie A.-G.; 595987 of Apr. 2, 1934 to Sudfeldt & Co.; **French Pats.** 576336 of Jan. 25, 1924 to M. V. de Bataafsche Petroleum Maatschappij; 593134 of Feb. 10, 1924 to Asphalt Cold Mix, Ltd.; **Dutch Pats.** 14751 of Jun.

10, 1926 to N. V. de Bataafsche Petroleum Maatschappij; 17050 of Jul. 4, 1925 to N. V. Koninklijke Stearin Kaarsenfabriek Gouda; **Hungarian Pat.** 103128 of Nov. 3, 1928 to Zsigmond Gádor.

55 [p. 510] **Can. Pat.** 326235 of Sep. 20, 1932 to Twitchell Process Co.

56 [p. 510] **U. S. Pat.** 1882741 of Oct. 18, 1932 to Hans Beller and Martin Luther; **French Pat.** 674215 of Apr. 29, 1929 to I. G. Farbenindustrie, A.-G.

57 [p. 510] **Ger. Pat.** 564922 of Mar. 21, 1931 to I. G. Farbenindustrie, A.-G.

58 [p. 510] **Ger. Pat.** 429445 of Aug. 5, 1924 to Gesellschaft für chemische Industrie; **French Pat.** 673887 of Apr. 23, 1929 to I. G. Farbenindustrie, A.-G.; **Swiss Pat.** 111990 of Aug. 2, 1924 to Gesellschaft für chemische Industrie.

59 [p. 510] **U. S. Pats.** 1747653 of Feb. 18, 1930 to Charlton Wilder; 1772999 of Aug. 12, 1930 to F. W. Huber.

60 [p. 510] **U. S. Pats.** 2025945 of Dec. 31, 1935 to C. N. Forrest; 2026614 of Jan. 7, 1936 to J. H. Conzelman; 2041234 of May 19, 1936 to Karl Daimler; **Brit. Pat.** 210343 of May 23, 1923 to C. L. V. Zimmer and E. Fränkl; 379915 of Nov. 13, 1931 to N. V. Mijnbouw-en Cultuurmaatschappij "Boeton"; 452998 of Sep. 3, 1936 to S. A. Giovanni della Coletta; **Ger. Pat.** 562699 of Dec. 18, 1930 to N. V. Mijnbouw-en Cultuurmaatschappij "Boeton"; Appl. J-48326 of Nov. 11, 1935 to I. G. Farbenindustrie A.-G.; Appl. B-161603 of Jul. 11, 1933 to Barber Co., Inc.; **French Pats.** 726310 of Nov. 17, 1931 to N. V. Mijnbouw-en Cultuurmaatschappij "Boeton"; 727328 of Dec. 2, 1931 to W. R. Roederer.

61 [p. 510] **U. S. Pat.** 2083572 of Apr. 28, 1936 to Hans Klein; **Brit. Pats.** 395384 of Dec. 3, 1931 to W. R. Roederer; 402891 of Aug. 15, 1932 to W. R. Roederer; **Ger. Pat.** 608431 of Dec. 4, 1930 to W. R. Roederer; 615463 of Aug. 14, 1931 to W. R. Roederer.

62 [p. 510] **Ger. Pats.** Appl. R-494.30 of Dec. 3, 1930 to W. R. Roederer; Appl. R-82482 of Aug. 13, 1931 to W. R. Roederer.

63 [p. 510] **U. S. Pat.** 1609308 of Dec. 7, 1926 to W. B. Pratt; **Ger. Pat.** 564780 of Jul. 12, 1931 to Chem. Fabrik von Heyden, A.-G.

64 [p. 510] **U. S. Pats.** 884878 of Apr. 14, 1908 to J. P. Van Der Ploeg; 956009 of Apr. 26, 1910 to L. S. Van Westrum; 1778239 and 1778240 of Oct. 14, 1930 to W. J. Yates; 1854348 of Apr. 19, 1932 to Gustav Rodenwald; **Brit. Pats.** of 1905 (Jun. 2), 11620 to R. M. Hahn; 229361 of Sep. 19, 1923 to H. A. Mackay; 255044 of Jun. 22, 1926 to Paul Lechler; 300821 of Dec. 24, 1927 to J. A. Montgomerie; 332897 of Mar. 20, 1929 to C. G. Fox; **Ger. Pats.** 191399 of Apr. 8, 1905 to J. P. Van Der Ploeg; 363374 of Jul. 8, 1921 to Sanitol-Werke, G.m.b.H.; 549330 of Mar. 19, 1929 to Amber Size & Chemical Co., Ltd.; **Swiss Pat.** 148719 of May 1, 1930 to Romag A.-G. für Rohmaterialen.

65 [p. 510] **Brit. Pat.** 350744 of Jun. 24, 1930 to W. A. Walmsley and R. H. Thompson.

66 [p. 510] **U. S. Pats.** 786348 of Apr. 4, 1905 to L. A. Dreyfus; 788043 of Apr. 25, 1905 to F. X. Govers; 788857 of May 2, 1905 to G. A. Thubé and Louis Préaubert; 790821 of May 23, 1905 to H. V. Dunham; 812593 of Feb. 13, 1906 to Louis Préaubert and G. A. Thubé; 1010210 of Nov. 28, 1911 to C. L. V. Zimmer; 1357688 of Nov. 2, 1920 to Henry Chislet; 1432742 of Oct. 24, 1922 to F. J. Commin; 1440356 of Dec. 26, 1922 to J. C. Morrell; 1700581 of Jan. 29, 1929 to W. E. Billinghame; 1859517 of May 24, 1932 to Lester Kirschbraun; 2040115 of May 12, 1936 to V. E. Watts; 2074731 of Mar. 23, 1937 to C. L. McKesson; **Can. Pats.** 229000 of Feb. 27, 1923 to F. J. Commin; 247347 of Mar. 3, 1925 to N. V. Koninklijke Stearin Kaarsenfabriek Gouda; 255518 of Nov. 17, 1925 to Asphalt Cold Mix, Ltd.; 261175 of May 25, 1926 to Tarkold, Ltd.; 286384 of Jan. 15, 1929 to W. E. Billinghame; 325450 of Aug. 30, 1932 to Miroslav Hubmajer; 330943 of Mar. 14, 1933 to International Bitumen Emulsions Corp.; 342801 of Jul. 3, 1934 to International Bitumen Emulsions Corp.; **Brit. Pats.** of 1903 (May 14), 11028 to Louis Préaubert and G. A. Thubé; of 1905 (May 5), 9422 to Louis Préaubert and G. A. Thubé; 155398 of Sep. 30, 1919 to E. Schou; 221380 of Nov. 7, 1923 to L. W. Low; 222602 of Nov. 19, 1923 to W. E. Billinghame; 225587 of Jun. 6, 1923 to Knud Erslev; 230177 of Dec. 5, 1923 to H. A. Mackay; 251098 of Apr. 28, 1925 to W. J. McGivern, J. H. Foster and R. Swift; 252260 of Feb. 24, 1925 to G. S. Hay; 255074 of Jul. 7, 1926 to Georges Baume, Pierre Chambige and de Boutier; 255911 of Jul. 23, 1926 to W. E. Billinghame; 258870 of Sep. 20, 1926 to E. B. Hack; 286522 of Aug. 2, 1927 to N. V. Konink-

lijke Stearin Kaarsenfabriek Gouda; 301805 of Dec. 3, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 308389 of Jan. 7, 1928 to T. M. Hickman; 333303 of May 14, 1929 to Jonathan Parker; 365586 of Aug. 15, 1930 to Ragnvald Hellerud; 374111 of Mar. 4, 1931 to J. A. Montgomerie; 393868 of Nov. 19, 1932 to C. G. Fox and S. Stockwell; 401131 of Nov. 9, 1933 to International Bitumen Emulsions Corp.; **Ger. Pats.** 240482 of May 1, 1910 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 342639 of Dec. 24, 1920 to F. J. Commin; 405930 of Nov. 22, 1922 to Ernst Stern; 418107 of Oct. 23, 1924 to Tarkold, Ltd.; 514399 of Jul. 17, 1927 to Hermann Bollmann and Bruno Rewald; 516187 of Sep. 7, 1927 to Hermann Bollmann and Bruno Rewald; 516188 of Oct. 25, 1927 to Hermann Bollmann and Bruno Rewald; 516189 of Dec. 25, 1927 to Hermann Bollmann and Bruno Rewald; 643350 of Aug. 28, 1931 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger A.-G.; Appl. G-91234 of Sep. 9, 1935 to Th. Goldschmidt A.-G.; **French Pats.** 529295 of Dec. 31, 1920 to F. J. Commin; 606535 of Nov. 11, 1925 to W. E. Billinghame; 623777 of Oct. 28, 1926 to G. Murphy; 732463 of Feb. 29, 1932 to Bitumen Investments, Inc.; **Swiss Pat.** 112803 of Sep. 17, 1924 to Asphalt Cold Mix, Ltd.

67 [p. 510] **U. S. Pat.** 1855934 of Apr. 26, 1932 to H. P. Banks, Glenn Davidson, I. F. Laucks and H. F. Rippey; 2020662 of Nov. 12, 1935 to Albert Schweiger; **Ger. Pat.** 522041 of Dec. 6, 1927 to Hanseatische Mühlenwerke, A.-G.; 604328 of Dec. 2, 1932 to Deutsche Vitalit-Gesellschaft, m.b.H.; **French Pat.** 730961 of Feb. 3, 1932 to Gesellschaft für Teerverwertung, m.b.H.

68 [p. 510] **U. S. Pat.** 1950272 of Mar. 6, 1934 to A. E. Schutte.

69 [p. 510] **U. S. Pats.** 1440355 and 1440356 of Dec. 26, 1922 to J. C. Morrell; 1755379 of Apr. 22, 1930 to H. W. Banks; 1960115 of May 22, 1934 to Z. C. Loebel; 1969659 of Aug. 7, 1934 to W. W. McLaurin; **Can. Pats.** 313274 of Jul. 14, 1931 to Flintkote Roads, Inc.; 333343 of Jun. 20, 1933 to W. W. McLaurin; **Brit. Pats.** 255074 of Jul. 7, 1926 to Georges Baume; 274142 of Jan. 15, 1926 to J. H. Disney and J. C. Kernot; 322792 and 322793 of Sep. 13, 1928 to Colas Products Ltd. and L. G. Gabriel and J. F. Blott; 323060 of Oct. 6, 1928 to F. N. Nicholls; **Ger. Pats.** 127582 of Jun. 17, 1900 to Carl Baswitz; 312690 of May 10, 1912 to W. Plinatus; 421237 of May 3, 1924 to A. Knecht.

70 [p. 510] **U. S. Pat.** 1647805 of Nov. 1, 1927 to John McGavack; **Brit. Pats.** 221466 of Feb. 27, 1924 to Douglas Pectin Corp.; 276878 of Oct. 5, 1926 to California Fruit Growers' Exchange; **Ger. Pat.** 384772 of Apr. 11, 1917 to Berlin Dextrinfabrik, Otto Kutzner; 560259 of Feb. 7, 1932 to Deutsche Pektingesellschaft m.b.H.

71 [p. 510] **U. S. Pats.** 834830 of Oct. 30, 1906 to Karl Mann; 943667 of Dec. 21, 1909 to Carleton Ellis; 1609308 of Dec. 7, 1926 to W. B. Pratt; 1754535 of Apr. 15, 1930 to W. B. Wescott; 1831492 of Nov. 10, 1931 to E. A. Hauser; 1869526 of Aug. 2, 1932 to M. L. Tower, H. W. Dye and F. L. McDonough; **Brit. Pats.** 239120 of May 31, 1924 to G. S. Hay; 255074 of Jul. 7, 1926 to Georges Baume, Pierre Chambige and de Boutier; 286527 of Jun. 15, 1927 to Johnson & Johnson; 337269 of Jan. 24, 1929 to K. D. P., Ltd.; 351242 of Jun. 26, 1930 to F. J. E. China and W. A. White; 405906 of Feb. 15, 1934 to W. H. Wilcken; **Ger. Pat.** 127852 of Jan. 18, 1902 to Carl Baswitz; **French Pat.** 686821 of Dec. 18, 1929 to K. D. P., Ltd.; 760958 of Mar. 7, 1934 to W. H. Wilcken.

72 [p. 510] **French Pat.** 633687 of Sep. 6, 1926 to P. Gloess and M. Marini.

73 [p. 510] **U. S. Pat.** 1653026 of Dec. 20, 1927 to F. C. Thornley, F. F. Tapping and Otto Reynard; **Can. Pat.** 252659 of Aug. 11, 1925 to F. C. Thornley and F. F. Tapping; 338571 of Jan. 9, 1934 to G. S. Rutherford; **Brit. Pat.** 219348 of Jan. 26, 1923 to F. C. Thornley and F. F. Tapping; 294002 of Aug. 25, 1927 to General Rubber Co.; **French Pats.** 583759 of Jul. 15, 1924 to F. C. Thornley; 618918 of Jul. 13, 1926 to Chem. Fabr. Dr. Joachim Wiernick.

74 [p. 510] **U. S. Pats.** 1582467 of Apr. 27, 1926 to G. S. Hay; 1738022 of Dec. 3, 1929 to J. P. Strasser; 1769629 of Jul. 1, 1930 to A. C. Fischer; 1881729 of Oct. 11, 1932 to H. L. Levin; 2044570 of Jun. 16, 1936 to C. E. Hite; 2047258 of Jul. 14, 1936 to Maurice Ernotte; **Can. Pats.** 264635 of Sep. 28, 1926 to Asphalt Cold Mix, Ltd.; 266901 of Dec. 21, 1926 to Asphalt Cold Mix, Ltd.; 349595 of Apr. 16, 1935 to Flintkote Co.; **Brit. Pats.** 243398 of May 31, 1924 to G. S. Hay; 248859 of Dec. 16, 1924 to G. S. Hay; 305742 of Nov. 26, 1927 to

E. C. R. Marks; 344490 of Jul. 9, 1929 to Union Chimique Belge, S. A.; 346978 of Apr. 24, 1930 to Nils Bendixen and J. G. Y. D. Morgan; 419358 of Apr. 5, 1933 to Eugéne Rouault; **Ger. Pat.** 170133 of Jun. 3, 1904 to Karl Mann; 590958 of Apr. 16, 1932 to I. G. Farbenindustrie, A.-G.; Appl. K–128183 of Dec. 13, 1932 to Rudolf Köhler and Hans Erbring; Appl. E–45516 of Apr. 17, 1934 to Maurice Ernotte; **French Pats.** 591040 of Dec. 27, 1924 to Asphalt Cold Mix, Ltd.; 748886 of Jul. 12, 1933 to Eugéne Rouault; **Danish Pat.** 33864 of Mar. 14, 1924 to H. Lange; **Japanese Pat.** 91327 of May 9, 1931 to Keita Maki.

75 [p. 510] "Dust Preventatives and Road Binders," by Prévost Hubbard, pp. 109 et seq. John Wiley & Sons, New York (1910); **U. S. Pats.** 62810 of Mar. 12, 1867 to A. F. Boon; 349751 of Sep. 28, 1886 to A. H. Rowand and R. S. Hunzeker; **Ger. Pat.** 61555 of Apr. 4, 1892 to Georg Friedrich Co.; 632015 of Jul. 1, 1936 to Maurice Ernotte; **Austrian Pat.** 101032 of Mar. 27, 1923 to A. Neumann; **Belgian Pats.** 395761 of May 31, 1933 to Maurice Ernotte; 402135 of Apr. 30, 1934 to Maurice Ernotte.

76 [p. 510] **U. S. Pats.** 1440356 of Dec. 26, 1922 to J. C. Morrell; 1609308 of Dec. 7, 1926 to W. B. Pratt; 1832987 of Nov. 24, 1931 to Lester Kirschbraun; 1840157 of Jan. 5, 1932 to C. F. Cross and Alf Engelstad; 1878828 of Sep. 20, 1932 to C. F. Cross and Alf Engelstad; 1988543 of Jan. 22, 1935 to Karl Daimler; 2002505 of May 28, 1935 to A. W. Hixson and J. M. Fain; 2008978 of Jul. 23, 1935 to Karl Daimler; **Can. Pats.** 262783 of Jul. 20, 1926 to Asphalt Cold Mix, Ltd.; 288853 of Apr. 16, 1929 to I. G. Farbenindustrie, A.-G.; **Brit. Pat.** 246907 of Nov. 3, 1924 to Asphalt Cold Mix, Ltd. and Frank Levy; 263520 of Sep. 21, 1925 to C. F. Cross and Alf Engelstad; 317496 of Mar. 16, 1928 to I. G. Farbenindustrie, A.-G.; 334426 of Oct. 20, 1928 to I. G. Farbenindustrie, A.-G.; 351904 of Dec. 15, 1928 to I. G. Farbenindustrie, A.-G.; 362467 of Jul. 5, 1930 to I. G. Farbenindustrie, A.-G.; 366158 of Nov. 7, 1930 to I. G. Farbenindustrie, A.-G.; 366174 of Nov. 17, 1930 to I. G. Farbenindustrie, A.-G.; 367782 of Nov. 18, 1930 to I. G. Farbenindustrie, A.-G.; 381933 of May 27, 1931 to I. G. Farbenindustrie, A.-G.; 430917 of Dec. 27, 1933 to W. W. Groves; 433550 of Feb. 16, 1934 to W. W. Groves; 441879 of Jul. 27, 1934 to W. W. Groves. **Ger. Pats.** 313339 of Mar. 17, 1914 to W. Riese; 390434 of Mar. 28, 1922 to Plauson's Forschungsinstitut, G.m.b.H.; 562050 of Apr. 15, 1931 to I. G. Farbenindustrie, A.-G.; 591340 of Dec. 19, 1929 to Gesellschaft für Teerverwertung, m.b.H.; 627465 of Nov. 8, 1933 to I. G. Farbenindustrie, A.-G.; 629270 of Apr. 25, 1936 to I. G. Farbenindustrie, A.-G.; **French Pats.** 597925 of Aug. 14, 1924 to Soc. de Recherches; 606418 of Nov. 3, 1925 to Asphalt Cold Mix, Ltd.; 705797 of Nov. 15, 1930 to I. G. Farbenindustrie, A.-G.; 707505 of Dec. 13, 1930 to I. G. Farbenindustrie, A.-G.; 725296 of Jan. 7, 1931 to Soc. des Produits Chimiques et Matières Colorantes de Mulhouse.

77 [p. 510] **Brit. Pat.** of 1905 (Jun. 2), 11620 to R. M. Hahn.

78 [p. 510] **U. S. Pat.** 1997868 of Apr. 16, 1935 to H. L. Levin.

79 [p. 510] **U. S. Pat.** 1973991 of Sep. 18, 1934 to C. L. McKesson, L. G. Thompson and W. D. Buckley.

80 [p. 510] **U. S. Pat.** 2005113 of Jun. 18, 1935 to Orvall Smiley; **Can. Pat.** 328800 of Dec. 27, 1932 to Orvall Smiley.

81 [p. 511] **U. S. Pats.** 1305645 of Jun. 3, 1919 to Samuel Cabot; 1783366 of Dec. 2, 1930 to R. W. Lewis; 1957408 of May 1, 1934 to Eugen Hutzenlaub; **Can. Pat.** 297226 of Feb. 4, 1930 to J. A. Montgomerie; **Brit. Pats.** 321334 of Nov. 17, 1928 to J. A. Montgomerie; 351073 of May 9, 1930, to C. M. Cunynghame and C. W. Fulton; 428571 of Aug. 1, 1934 to G. M. Skinner; Ltd.; 436494 of Mar. 18, 1935 to F. V. Lister.

82 [p. 511] **U. S. Pat.** 1738776 of Dec. 10, 1929 to Lester Kirschbraun; 2033657 of Mar. 10, 1936 to P. R. Smith; **Brit. Pat.** 436494 of Oct. 11, 1935 to F. V. Lister.

83 [p. 511] **U. S. Pat.** 1788706 of Jan. 13, 1931 to Roy Cross.

84 [p. 511] **U. S. Pat.** 1733494 of Oct. 29, 1929 to Lester Kirschbraun; **French Pat.** 705505 of Nov. 5, 1930 to S. W. Aretz.

85 [p. 511] **U. S. Pats.** 58975 of Oct. 23, 1866 to Franklin Bearse and G. E. Hopkins; 1733495 and 1733496 of Oct. 29, 1929 to Lester Kirschbraun; **Can. Pat.** 313276 of Jul. 14, 1931 to Flintkote Co.; **Brit. Pats.** 312467 of May 4, 1928 to F. W. Gough; 341443 of Oct. 9, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

86 [p. 511] **U. S. Pat.** 1408224 of Feb. 28, 1922 to C. S. Reeve; **Ger. Pat.** 601891 of Aug. 18, 1931 to Paul Lechler; **French Pat.** 529956 of Feb. 6, 1925 to F. Simon.

87 [p. 511] **U. S. Pats.** 1469563 of Oct. 2, 1923 to Lester Kirschbraun; 1517075 of Nov. 25, 1924 to Lester Kirschbraun; 1700581 of Jan. 29, 1929 to W. E. Billinghame; 1733493 of Oct. 29, 1929 to Lester Kirschbraun; 1752449 of Apr. 1, 1930 to J. S. Miller; 1793918 of Feb. 24, 1931 to J. M. Fain; 1884919 of Oct. 25, 1932 to L. G. Thompson; 1913430 of Jun. 13, 1933 to O. E. Cushman; 1948881 of Feb. 27, 1934 to Lester Kirschbraun; 2027582 of Jan. 14, 1936 to J. M. Fain; 2061076 of Nov. 17, 1936 to Lester Kirschbraun; **Can. Pats.** 279241 of Apr. 10, 1928 to Lester Kirschbraun; 310872 and 310873 of Apr. 28, 1931 to Flintkote Roads, Inc.; 315495 of Apr. 25, 1928 to G. C. Hurrell; **Brit. Pats.** 312580 of May 27, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 342031 of Nov. 15, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 392365 of May 18, 1933 to Paul Lechler; 400409 of Oct. 13, 1932 to F. B. Dehn; **Ger. Pat.** 569499 of May 18, 1929 to N. V. de Bataafsche Petroleum Maatschappij; **French Pats.** 684531 of Nov. 7, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 690242 of May 22, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 738596 of Jun. 14, 1932 to Paul Lechler; **Hungarian Pat.** 101550 of Oct. 12, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

88 [p. 511] **U. S. Pat.** 1561728 of Nov. 17, 1925 to Lester Kirschbraun.

89 [p. 511] **U. S. Pats.** 1700581 of Jan. 29, 1929 to W. E. Billinghame; 1734437 of Nov. 5, 1929 to Lester Kirschbraun; 1859517 of May 24, 1932 to Lester Kirschbraun.

90 [p. 511] **U. S. Pat.** 1440356 of Dec. 26, 1922 to J. C. Morrell.

91 [p. 511] **U. S. Pat.** 1733498 of Oct. 29, 1929 to Lester Kirschbraun.

92 [p. 511] **U. S. Pats.** 1440356 of Dec. 26, 1922 to J. C. Morrell; 1733498 of Oct. 29, 1929 to Lester Kirschbraun; 1869697 of Aug. 2, 1932 to Lester Kirschbraun.

93 [p. 511] **Ger. Pat.** Appl. C–160.30 of Oct. 22, 1930 to Chemieprodukte, G.m.b.H.

94 [p. 511] **Ger. Pat.** 583237 of Jun. 2, 1931 to Robert Tagg; Appl. T–38952 of Jun. 1, 1931 to Robert Tagg.

95 [p. 511] **Brit. Pat.** 344562 of Dec. 18, 1929 to J. Leben and Ormul Products, Ltd.

96 [p. 511] **U. S. Pat.** 1956779 of May 1, 1934 to J. W. Sparks.

97 [p. 511] **U. S. Pat.** 1956779 of May 1, 1934 to J. W. Sparks; **Brit. Pat.** 341914 of Oct. 21, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

98 [p. 511] **Swiss Pat.** 122055 of Feb. 12, 1926 to Mineral A.-G.

99 [p. 511] **U. S. Pat.** 1925672 of Sep. 5, 1933 to A. A. Oeding.

100 [p. 511] **U. S. Pat.** 1831544 of Nov. 11, 1931 to W. B. Pratt and R. J. Noble.

101 [p. 511] **U. S. Pat.** 1977558 of Oct. 16, 1934 to R. K. Oschatz; 2022229 of Nov. 26, 1935 to U. B. Bray and L. B. Beckwith; **Brit. Pats.** 379076 of Aug. 6, 1931 to Chemische und Seifenfabrik R. Baumheier A.-G.; 400045 of Oct. 19, 1933 to R. J. Lacau; **Ger. Pat.** 567776 of May 10, 1927 to W. A. Whatmough.

102 [p. 511] **U. S. Pat.** 1867923 of Jul. 19, 1932 to Frederic Queck; **Brit. Pat.** 320847 of May 22, 1928 to Colas Products, Ltd.; **Ger. Pats.** 594310 of Jul. 10, 1926 to Georges Baume, Pierre Chambige and Denis Boutier; 603379 of Nov. 12, 1927 to Georges Baume, Pierre Chambige and Denis Boutier.

103 [p. 511] **Brit. Pat.** 430061 of Jun. 8, 1934 to Soc. de Recherches et de Perfect. Industr.; **French Pat.** 701934 of Sep. 13, 1930 to F. J. E. China, W. A. White and Burt, Boulton & Haywood, Ltd.

104 [p. 511] **U. S. Pat.** 1932648 of Oct. 31, 1933 to Kenneth Taylor; 2022229 of Nov. 26, 1935 to U. B. Bray and L. B. Beckwith; **Brit. Pat.** 419358 of Apr. 5, 1933 to Eugéne Rouault; **Ger. Pat.** Appl. R–87697 of Apr. 5, 1933 to Eugéne Rouault.

105 [p. 511] **U. S. Pats.** 1869379 and 1869380 of Aug. 2, 1932 to R. W. Lewis.

106 [p. 511] **U. S. Pat.** 1010210 of Nov. 28, 1911 to K. L. V. Zimmer.

107 [p. 511] **U. S. Pat.** 1873580 of Aug. 23, 1932 to A. J. Hailwood, Arnold Shepherdson and Alexander Stewart; **Can. Pat.** 305469 of Nov. 4, 1930 to Imperial Chemical Industries, Ltd.

108 [p. 511] **U. S. Pat.** 1816018 of Jul. 28, 1931 to C. C. Loomis and H. E. Stump.

109 [p. 511] "Stabilization of Aqueous Dispersions of Asphalt," by A. W. Hixson and J. M. Fain, *J. Ind. Eng. Chem.*, **24**, 1336 (1932).

110 [p. 511] **Brit. Pat.** 301805 of Dec. 3, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 380052, 380065 and 380076 of Sep. 1, 1932 to Erba Fabrik chemischer Produkte.

111 [p. 511] **U. S. Pat.** 1086235 of Feb. 3, 1914 to I. S. Stahl.

112 [p. 511] **U. S. Pat.** 1878974 of Sep. 20, 1932 to Lester Kirschbraun.

113 [p. 511] **U. S. Pat.** 1995346 of Mar. 26, 1935 to A. L. Halvorsen.

114 [p. 511] **U. S. Pat.** 2009821 of Jul. 30, 1935 to A. G. Terry, L. G. Gabriel and J. F. T. Blott; **Brit. Pat.** 312580 of May 26, 1928 to Lester Kirschbraun. **Ger. Pat.** 569499 of May 18, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

115 [p. 511] "Three Phase Emulsions," by H. Bechhold, L. Dede and L. Reiner, *Kolloid Zeits.*, 28, 6 (1921).

116 [p. 512] **U. S. Pat.** 1989374 of Jan. 29, 1935 to K. E. McConnaughay; 2042038 of May 26, 1936 to R. I. Colmar; **Brit. Pat.** 362299 of Apr. 2, 1931 to C. G. J. Lefebvre and E. E. F. Berger; **Ger. Pat.** Appl. B–126378 of Jul. 9, 1926 to Georges Baume and Pierre Chambige; **French Pat.** 723728 of Dec. 12, 1930 to C. G. J. Lefebvre and E. E. F. Berger.

117 [p. 512] **Ger. Pat.** 489476 of Jan. 2, 1926 to C. A. Braun.

118 [p. 512] **Brit. Pats.** 322792 of Sep. 13, 1928 to Colas Products, Ltd., L. G. Gabriel and J. F. Blott; 324663 of Oct. 24, 1928 to N. V. de Bataafsche Petroleum Maatschappij and H. D. Elkington; **Ger. Pat.** 579125 of Aug. 28, 1928 to N. V. de Bataafsche Petroleum Maatschappij.

119 [p. 512] **U. S. Pats.** 1984023 and 1984024 of Dec. 11, 1934 to Hyman Limberg; **Brit. Pat.** 350040 of Mar. 11, 1930 to H. D. Elkington; **Ger. Pat.** 557677 of Mar. 13, 1930 to Flintkote Co.

120 [p. 512] **Ger. Pat.** 542239 of Sep. 22, 1929 to Mitteldeutsche Teerprodukten- und Dachpappen-Fabrik.

121 [p. 512] **U. S. Pats.** 1010209 of Nov. 28, 1911 to K. L. V. Zimmer; 1963231 of Jun. 19, 1934 to J. M. Fain; 2046902 of Jul. 7, 1936 to Lester Kirschbraun; **Can. Pat.** 313276 of Jul. 14, 1931 to Flintkote Co.; **Brit. Pats.** 322923 of Jan. 9, 1929 to Berry, Wiggins & Co., Ltd.; 324663 of Jan. 31, 1930 to H. D. Elkington; **Ger. Pats.** 256573 of Feb. 15, 1910 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 367476 of Mar. 31, 1921 to Wilhelm Reiner; 491844 of Mar. 6, 1928 to Wilhelm Reiner; 546517 of Jul. 31, 1927 to Wilhelm Ackermann; 571384 of Oct. 18, 1929 to N. V. de Bataafsche Petroleum Maatschappij; **French Pat.** 760544 of Feb. 24, 1934 to Bitumen Investments, Inc.; **Austrian Pat.** 147164 of Oct. 10, 1936 to "Emulgia" Komm.-Ges. Clauser & Co. für Asphalt, Teer und Strassenbau.

122 [p. 512] **U. S. Pat.** 1918759 of Jul. 18, 1933 to Lester Kirschbraun; **Can. Pat.** 318061 of Dec. 15, 1931 to Flintkote Co.; **Ger. Pat.** 546517 of Jul. 31, 1927 to Wilhelm Ackermann.

123 [p. 512] **U. S. Pat.** 2027584 of Jan. 14, 1936 to L. G. Gabriel, J. F. T. Blott, W. L. Peard and L. W. G. Firmin; **Brit. Pat.** 389810 of Mar. 20, 1933 to Colas Products, Ltd.; **French Pat.** 738823 of Jun. 18, 1932 to Colas Flintkote, Ltd.

124 [p. 512] **U. S. Pats.** 1737491 of Nov. 26, 1929 to C. A. Braun; 2009821 of Jul. 30, 1935 to A. G. Terry, L. G. Gabriel and J. F. T. Blott; 2055612 of Sep. 29, 1936 to Thomas Robinson; **Can. Pat.** 345814 of Nov. 6, 1934 to Barrett Co.; **Brit. Pats.** 254012 of Mar. 25, 1925 to C. A. Braun and G. S. Hay; 254701 of Jul. 1, 1925 to Paul Lechler; 362577 of Oct. 2, 1930 to Colas Products, Ltd.; 438680 of Nov. 21, 1935 to Lancaster Processes, Inc.; 441537 of Oct. 12, 1934 to Lancaster Processes, Inc.; **Ger. Pats.** 514485 of Mar. 22, 1925 to Bitumuls Kaltasphalt A.-G.; Appl. A–21194 of Sep. 25, 1911 to Kurt Albert and Ludwig Berend (rejected).

125 [p. 512] **U. S. Pats.** 1699536 and 1699537 of Jan. 22, 1929 to L. C. Jones, C. C. Loomis and H. W. Banks; 1725648 of Aug. 20, 1929 to Lester Kirschbraun; **Can. Pat.** 280289 of May 22, 1928 to Lester Kirschbraun; **Brit. Pats.** 255074 of Jul. 7, 1926 to Georges Baume, Pierre Chambige and de Boutier; 269155 of Mar. 24, 1927 to I. G. Farbenindustrie, A.-G.; 334426 of Oct. 21, 1929 to I. G. Farbenindustrie, A.-G.; **Ger. Pats.** 52129 of May 8, 1889 to W. Dammann; 321113 of Jul. 3, 1917 to E. Stephani; 337769 of Jan. 30, 1920 to Max Rogler; Appl. A–58456 of Jul. 13, 1929 to C. A. Agthe.

126 [p. 512] **Brit. Pat.** 333152 of Feb. 4, 1929 to C. G. Fox.

127 [p. 512] **U. S. Pat.** 1778760 of Oct. 21, 1930 to G. S. Hay; **Can. Pat.** 266901 of Dec. 21, 1926 to Asphalt Cold Mix, Ltd.; **Brit. Pat.** 255684 of Oct. 1, 1925 to W. J. McGivern;

Ger. Pat. 363374 of Jul. 8, 1921 to Sanitol-Werke, G.m.b.H.; **French Pat.** 651794 of Mar. 26, 1928 to Arnaldo Caroselli and Heinrich Heinz.

128 [p. 512] **Brit. Pat.** 255684 of Oct. 1, 1925 to W. J. McGivern; **French Pat.** 698859 of Jul. 11, 1930 to C. A. Agthe.

129 [p. 512] **U. S. Pat.** 1778760 of Oct. 21, 1930 to G. S. Hay; **Can. Pat.** 300450 of May 20, 1930 to F. W. Atack; **Brit. Pat.** 277356 of Sep. 8, 1927 to Colas Cold Asphalt Co.

130 [p. 512] **Brit. Pat.** 319101 of Jul. 25, 1928 to Hermann Plauson.

131 [p. 512] **Japan Pat.** 96313 of Jun. 17, 1932 to Yosimasa Itikawa and Sinken Maki.

132 [p. 513] "Bituminized Cement," by G. Knapp, *Schweiz Z. Strassenw.*, 21, 265 (1935); "Standard Specifications for Curing Portland-Cement Concrete Slabs with Bituminous Coverings" (C 81–36), A.S.T.M. Standards 1936, II, 324; **U. S. Pats.** 1690020 of Oct. 30, 1928 to Lester Kirschbraun; 1862011 of Jun. 7, 1932 to R. B. Gage; 2000759 of May 7, 1935 to A. A. Johnson; **Can. Pats.** 256079 of Dec. 8, 1925 to Lester Kirschbraun; 289974 of May 28, 1929 to Barber Asphalt Co.; 313273 of Jul. 14, 1931 to Flintkote Roads, Inc.; 325519 of Aug. 30, 1932 to Colas Roads, Inc.; 325999 of Sep. 13, 1932 to International Bitumen Emulsions Corp.; **Brit. Pat.** 375460 of Apr. 7, 1931 to G. A. Lasseur and T. V. Taralou.

133 [p. 513] "Technology of Emulsions for Surfacing Floors," by Lester Kirschbraun, *Eng. News-Record*, 114, 276 (1935); **U. S. Pats.** 1302810 of May 6, 1919 to Lester Kirschbraun; 1767533 of Jun. 24, 1930 to Lester Kirschbraun; 1991755 of Feb. 19, 1935 to H. L. Levin; 2062615 of Dec. 1, 1936 to E. W. Scripture, Jr.; 2074758 of Mar. 23, 1937 to L. N. Reynolds; **Can. Pat.** 328849 of Dec. 27, 1932 to Flintkote Corp.; **Brit. Pats.** 366304 of Jun. 4, 1930 to Nicolaas van der Willik; 367161 of Feb. 6, 1931 to J. P. Spratling; **Ger. Pat.** 491844 of Mar. 6, 1928 to Wilhelm Reiner.

134 [p. 513] "Latex; Its Occurrence, Collection, Properties and Technical Applications," by E. A. Hauser, Chemical Catalog Co. Inc., New York (1930); "Latex and Its Industrial Applications," by Frederick Marchionna, The Rubber Age Publishing Co., New York (1933); "Rubber from Latex-Bitumen Mixtures," by G. Martin, W. S. Davey, H. C. Baker and E. Rhodes, J. Rubber Research Inst. Malaya, 5, 311 (1934); "Latex in Industry," by R. J. Noble, The Rubber Age, New York (1936); "Rubber Latex," by H. P. Stevens and W. H. Stevens, 4th Edition, Rubber Growers' Association, London (1936); **U. S. Pats.** 1417837 of May 30, 1922 to Lester Kirschbraun; 1498387 of Jun. 17, 1924 to Lester Kirschbraun; 1726473 of Aug. 27, 1929 to W. P. Davey; 1750767 of Mar. 18, 1930 to M. C. Teague; 1831226 of Nov. 10, 1931 to J. N. Byrd; 1861407 of May 31, 1932 to A. C. Fischer; 1872112 of Aug. 16, 1932 to A. E. Brown; 1833038 of Nov. 24, 1931 to W. B. Pratt; 1886334 of Nov. 1, 1932 to L. G. Gabriel and J. F. Blott; 1890476 of Dec. 13, 1932 to P. C. van der Willigen; 1909080 of May 16, 1933 to C. B. Strauch; 1911631 of May 30, 1933 to H. L. Levin; 1929499 of Oct. 10, 1933 to Lester Kirschbraun; 1950451, 1950452 and 1950453 of Mar. 13, 1934 to H. L. Levin; 1968459 of Jul. 31, 1934 to H. L. Levin; 1978022 of Oct. 23, 1934 to Lester Kirschbraun; 1988126 of Jan. 15, 1935 to Lester Kirschbraun; 2037948 of Apr. 21, 1936 to C. D. Svensson; **Can. Pats.** 294718 of Nov. 12, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 305679 of Nov. 11, 1930 to Flintkote Co.; 329640 of Jan. 24, 1933 to Flintkote Corp.; 330015 and 330016 of Feb. 7, 1933 to Flintkote Corp.; 331483 of Apr. 4, 1933 to Flintkote Corp.; **Brit. Pats.** 254004 of Mar. 25, 1925 to Frank Levy; 273031 of Apr. 6, 1926 to Universal Rubber Paviors; 278395 of Apr. 7, 1926 to N. V. de Bataafsche Petroleum Maatschappij; 301805 of Dec. 3, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 307466 of Mar. 8, 1929 to Kaspar Winkler; 312372 of Nov. 18, 1927 to G. Plauson; 315839 of Jul. 17, 1929 to Lester Kirschbraun; 329965 of Feb. 27, 1929 to Colas Products, Ltd., L. G. Gabriel and J. F. Blott; 337521 of Sep. 4, 1929 to J. Y. Johnson; 357603 of May 28, 1929 to S. S. Sadtler; 358142 of Jul. 10, 1930 to C. A. Agthe; 359924 of May 28, 1930 to S. S. Sadtler; 380703 of Feb. 3, 1932 to Flintkote Corp.; 383432 of Nov. 26, 1931 to G. W. Trobridge; 384138 of Feb. 18, 1932 to K. D. Svensson; 419629 of Sep. 29, 1933 to International Latex Processes, Ltd.; 428100 of May 7, 1935 to Rubber Producers' Research Association; 433741 of Aug. 20, 1935 to I. G. Farbenindustrie, A.-G.; 399684 of Jan. 5, 1933 to László d'Antal; 446814 of Aug. 3, 1934 to R. Riecke; 453299 of Jun. 13, 1936 to R. K. Maclean; **Ger. Pat.** 609008 of Feb. 6, 1935 to Adam Loew and Carl Halang; 611491 of

Mar. 29, 1935 to Wilhelm Reiner; **French Pats.** 577488 of Aug. 3, 1926 to Cie. française pour l'exploitation des Procédes Thompson-Houston; 605288 of Oct. 6, 1925 to J. M. A. Touchon; 648917 of Feb. 4, 1928 to Victor Wintsch, Jr.; 728324 of Dec. 16, 1931 to A. E. Bond, and William Andrews; 756535 of Dec. 11, 1933 to Gustave Labourse; 756535, Addition 44177 of Nov. 20, 1934 to Gustave Labourse; **Swedish Pat.** 86859 of Jul. 14, 1936 to Aktiebolaget Patentor; **Swiss Pat.** 177570 of Sep. 2, 1935 to G. H. Ott.

135 [p. 513] **U. S. Pat.** 1726708 of Sep. 3, 1929 to H. L. Levin; 2033869 and 2033870 of Mar. 10, 1936 to C. H. Smith.

136 [p. 513] **U. S. Pat.** 1914912 of Jun. 20, 1933 to G. B. Dunford; **Can. Pat.** 313896 of Aug, 4, 1931 to G. B. Dunford; **Brit. Pat.** 357514 of May 17, 1930 to G. B. Dunford; 450951 of Oct. 22, 1924 to I. G. Farbenindustrie A.-G.

137 [p. 513] **U. S. Pat.** 1984214 of Dec. 11, 1934 to M. J. Heitmann.

138 [p. 513] **U. S. Pat.** 1988175 of Jan. 15, 1935 to D. R. Merrill.

139 [p. 513] **U. S. Pat.** 2003921 of Jun. 4, 1935 to Louis Chevrillon; 2051505 of Aug. 18, 1936 to Jan van Hulst; 2075244 of Mar. 30, 1937 to Jan van Hulst; 2071758 of Feb. 23, 1937 to John McKay; **Can. Pat.** 364758 of Mar. 16, 1937 to Patent & Licensing Corp.; 365542 of Apr. 20, 1937 to Patent & Licensing Corp.; **Brit. Pats.** 456695 of Nov. 4, 1935 to N. V. de Bataafsche Petroleum Maatschappij; 456712 of Dec. 21, 1935 to N. V. de Bataafsche Petroleum Maatschappij.

140 [p. 513] **Can. Pat.** 350927 of Jun. 11, 1935 to Texaco Development Corp.

CHAPTER XXVI

1 [p. 516] "Lacquer Solvents in Commercial Use," by A. K. Doolittle, *J. Ind. Eng. Chem.*, **27**, 1169 (1935).

2 [p. 515] "Surface-Tension of Solutions," by G. N. Antonov, *J. chim. phys.*, **5**, 372 (1907).

3 [p. 521] "Solubility of Asphalt Bitumen and Asphaltenes in Mixed Solvents," by F. J. Nellensteyn, *Chem. Weekblad*, **28**, 313 (1931).

4 [p. 521] "Surface Tension-Temperature Curves of Asphalt Bitumens and Similar Products," by F. J. Nellensteyn and N. M. Roodenburg, *Kolloid-Chem. Beihefte*, **31**, 434 (1930); *Kolloidal Ztg.*, **63**, 339 (1933).

5 [p. 524] "Colloid Chemistry," by Jerome Alexander, Section entitled "Asphalt" by F. J. Nellensteyn, Vol. III, p. 535, Chemical Catalog Co., Inc., New York (1931).

6 [p. 525] "Determination of the Asphalt-bitumen Content in Asphalt-bitumen-tar Mixtures," by F. J. Nellensteyn and J. P. Kuipers, *Chem. Weekblad*, **29**, 291 (1932).

7 [p. 525] "Differentiation of Bituminous Materials," by H. Basler and J. Miegl, *Kolloid-Z.*, **59**, 345 (1932).

8 [p. 525] "Die Natürlichen und Künstlichen Asphalte und Peche," by E. J. Fischer, Verlag Theodor Steinkopff, Dresden and Leipzig, pp. 60–63, (1928); "Die Industrieteere, ihre Arten, Eigenschaften und Verwendung," by E. J. Fischer, *Teer u. Bitumen*, **27**, 17, 33, 69 and 125 (1929); "Künstliche Asphalte, Peche und ähnliche Produkte," by E. J. Fischer, *Teer u. Bitumen*, **28**, 17 and 33 (1930); "Organische Rohstoffe zur Herstellung technischer Asphalt- und Pechpräparate in ihrem Verhalten zu einigen weniger gebräuchlichen Lösungsmitteln," by E. J. Fischer, *Asphalt u. Teer*, **30**, 1930 (1930); "Lösungsverhältnisse von Asphalten und Pechen sowie nachweisverfahren für diese Stoffe," by E. J. Fischer, *Teer u. Bitumen*, **29**, 295 (1931); "Anwendung organischer Lösungsmittel bei der Analyse organisch-technischer Rohstoffe und Rohstoffgemische," by E. J. Fischer, Verlag von Wilhelm Knapp, Halle (Saale), (1932).

9 [p. 525] "The Adhesion of Bituminous Binders to Stone," by Walter Riedel and Hans Weber, *Asphalt u, Teer Strassenbautech.*, **33**, 677, 693, 713, 729, 749, 793 and 809 (1933); "The 'Breaking' of Bituminous Compositions upon Stone," by G. H. Klinkmann, *Asphalt u. Teer Strassenbautech.*, **33**, 842, 873 and 893 (1933).

10 [p. 525] "The Adsorption of Bitumen and its Constituents by Certain Natural and Treated Earths," by H. T. Lorne, *J. Inst. Petroleum Tech.*, **22**, 541 (1936).

CHAPTER XXVII

1 [p. 527] "Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level" (D 40–36T), A.S.T.M. Tentative Standards 1936, 817.

2 [p. 527] "Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level" (D 144–36T), A.S.T.M., Tentative Standards 1936, 815.

3 [p. 527] "Federal Spec. for Asphalt; (for) Built-up Roofing, Waterproofing, and Damp-proofing," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-A-666, Jun. 6, 1933.

4 [p. 527] **U. S. Pat.** 268260 of Nov. 28, 1882 to Duncan McLean.

5 [p. 527] **Ger. Pats.** 61555 of May 21, 1891 to Georg Friedrich & Co.; 65239 of Nov. 10, 1891 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 64680 of Sep. 23, 1891 to B. Roedelius; 73122 of Jan. 17, 1893 to C. Richard; 96094 of Mar. 11, 1896 to Richard Joecke; Appl. L–86365 of Aug. 7, 1934 to F. H. Lübben.

6 [p. 528] **Ger. Pat.** 325780 of Jul. 7, 1918 to Chem.-analyt. Lab. Ing. Schlick & Dr.-Ing. Horák.

7 [p. 528] **Brit. Pat.** 361084 of Oct. 27, 1930 to C. A. Agthe.

8 [p. 528] "Tentative Specifications for High-Carbon Coal-tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level" (D 42–36T), A.S.T.M. Tentative Standards 1936, 844.

9 [p. 528] "Tentative Specifications for High-Bitumen Coal-tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level" (D 200–36T), A.S.T.M. Tentative Standards 1936, 840.

10 [p. 528] "Tentative Specifications for High-Carbon Coal-tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level" (D 145–36T), A.S.T.M. Tentative Standards 1936, 842.

11 [p. 528] "Tentative Specifications for High-Bitumen Coal-tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level" (D 201–36T), A.S.T.M. Tentative Standards 1936, 838.

12 [p. 528] "Federal Spec. for Pitch; Coal-tar (for) Mineral-Surfaced Built-up Roofing, Waterproofing and Damp-proofing," Federal Standard Stock Catalogue, Section IV, (Part 5), R-P-381, Jun. 6, 1933, also Bureau of Standards Circular C–155.

13 [p. 529] "Tentative Specifications for Asphalt for Use in Constructing Built-up Roof Coverings" (D 312–35T) A.S.T.M. Tentative Standards 1936, 812.

14 [p. 529] "Federal Spec. for Asphalt; (for) Built-up Roofing, Waterproofing and Damp-proofing," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-A-666, Jun. 6, 1933.

15 [p. 530] "Asphalt-, Pech- und Teerkitte," by E. J. Fischer, *Teer u. Bitumen*, 34, 87 and 98 (1936).

16 [p. 530] **U. S. Pat.** 2011607 of Aug. 20, 1935 to C. S. Reeve.

17 [p. 530] "A Test Procedure for Plastic Caulking Materials," by D. W. Kessler, *Proc. A.S.T.M.*, 35, Part II, 581 (1935).

18 [p. 530] "Tentative Specifications for High-Carbon Coal-tar Pitch for Use in Constructing Built-up Roofs Sufaced with Slag or Gravel" (D 251–36T) A.S.T.M. Tentative Standards 1936, 836.

19 [p. 530] "Tentative Specifications for High-Bitumen Coal-tar Pitch for Use in Constructing Built-up Roofs Surfaced with Slag or Gravel" (D 252–36T), A.S.T.M. Tentative Standards 1936, 834.

20 [p. 530] "Federal Spec. for Pitch; Coal-tar (for) Mineral-Surfaced Built-up Roofing, Waterproofing and Damp-proofing," Federal Standard Stock Catalogue, Section IV, (Part 5), R-P-381; Jun. 6, 1933, also Bureau of Standards Circular C-157.

21 [p. 531] "Standard Specifications for Bituminous Grout for Use in Waterproofing Above Ground Level" (D 170–25), A.S.T.M. Standards 1936, II, 1127; "Standard Specifications for Bituminous Grout for Use in Waterproofing Below Ground Level" (D 171–25), A.S.T.M. Standards 1936, II, 1129.

22 [p. 531] **U. S. Pat.** 739820 of Sep. 29, 1903 to Henry Bolze; **Ger. Pat.** Design 1261682 of Feb. 15, 1933 to Wolfgang Fröhler.

23 [p. 531] "Tar Products for Water- and Damp-Proofing," by E. O. Rhodes and E. H. Hyde, *J. Ind. Eng. Chem.*, **27**, 1408 (1935).

24 [p. 531] "Protective Coatings for Shipbuilding Purposes; Bituminous Compositions," by H. H. Thayer, *Marine Eng.*, **28**, 369 (1923).

25 [p. 532] "Specs. for Bituminous Primer and Enamel," 52–P–17, issued by the U. S. Navy.

26 [p. 535] **U. S. Pat.** 1999325 of Apr. 30, 1935 to Heinrich Bosari-Fischer.

27 [p. 535] **U. S. Pat.** 520600 of May 29, 1894 to J. A. Just.

28 [p. 535] **U. S. Pat.** 2057020 of Oct. 13, 1936 to J. M. Fain and Adolph Barthel.

29 [p. 535] Linseed oil and bituminous paints, also inorganic materials such as alloys, oxides, silicates, and Portland-cement mortar or concrete are also used for this purpose.

30 [p. 536] **Ger. Pat.** 614302 of Nov. 28, 1931 to Vereinigte Stahlwerke A.-G.

31 [p. 536] "Iron Corrosion, Anti-Fouling and Anti-Corrosive Paints," by L. E. Andés, Scott-Greenwood & Co., London (1900); "Rustless Coatings," by M. P. Wood, 1st Edition, Chapter XII, John Wiley & Sons, New York (1904); "The Corrosion and Preservation of Iron and Steel," by A. S. Cushman and H. A. Gardner, McGraw-Hill Book Co., New York (1910); "The Comparative Values of Various Coatings and Coverings for the Prevention of Soil and Electrolytic Corrosion of Iron Pipe," by R. B. Harper, *Am. Gas Light J.*, **91**, 429, 528, 575, 625 and 667 (1909); "The Corrosion of Iron and Steel," by Alfred Sang, McGraw-Hill Book Co., New York (1910); "The Industrial and Artistic Technology of Paint and Varnish," by A. H. Sabin, 2nd Edition, Chapter XVIII, John Wiley & Sons, Inc., New York (1917); "First Progress Report on Bituminous Coating Materials as a Means of Protecting Pipe Against Soil Corrosion," by K. H. Logan and E. H. Berger, *J. Am. Petroleum Inst.*, **8**, 356 (1927); "Rostschutz und Rostschutzanstrich," by Hermann Suida and Heinrich Salvaterra, Verlag von Julius Springer, Vienna (1931); "Development of Specifications for Protection of Underground Pipes," by K. H. Logan, *J. Ind. Eng. Chem.*, **27**, 1354 (1935).

32 [p. 536] "The Corrosion and Protection of Metals," by A. H. Sexton, The Scientific Publishing Co., Manchester, England (1905).

33 [p. 537] "Pipe-Line Protection," by Stanley Gill, *J. Ind. Eng. Chem.*, **25**, 49 (1933).

34 [p. 537] **Brit. Pat.** of 1848 (Oct. 19), 12291 to R. Angus Smith.

35 [p. 537] **U. S. Pat.** 1773131 of Aug. 19, 1930 to C. R. Eckert; **Can. Pats.** 315429 of Sep. 22, 1931 to Barrett Co.; 316686 of Nov. 3, 1931 to Barrett Co.; **Brit. Pat.** 343265 of Jan. 1, 1930 to Barrett Co.

36 [p. 538] **U. S. Pat.** 1914897 of Jun. 20, 1933 to P. F. Schade; **Brit. Pat.** 415384 of Feb. 16, 1933 to Chemieprodukte, G.m.b.H.

37 [p. 538] "An Example of Long-time Service of Baked Enamel Coating on Water Pipe," by A. H. Sabin, *Proc. A.S.T.M.*, **32**, 666 (1932); **Ger. Pats.** Appl. V–27071 of Jul. 13, 1931 to Vereinigte Stahlwerke, A.-G.; Appl. V–27468 of Oct. 29, 1931 to Vereinigte Stahlwerke, A.-G.

38 [p. 538] **Brit. Pat.** 397267 of May 24, 1932 to Adele Thielmann.

39 [p. 538] **U. S. Pat.** 2061825 of Nov. 24, 1936 to E. P. Bly; **Brit. Pats.** 304288 of Jan. 17, 1929 to B. E. Monteux and P. L. Durand; 365584 of Jul. 18, 1930 to B. E. Monteux and P. L. Durand.

40 [p. 538] **U. S. Pats.** 1648475 of Nov. 8, 1927 to W. P. Darwin; 1786785 of Dec. 30, 1930 to A. W. Spaulding; 1839891 of Jan. 5, 1932 to A. G. Perkins; Reissue 18334 of Jan. 19, 1932 to W. P. Darwin; **Ger. Pats.** 528331 of Jan. 5, 1930 to Jakob Kuelers; Appl. St–46095 of Jul. 9, 1929 to Stewarts & Lloyds, Ltd.

41 [p. 538] **U. S. Pats.** 1572258 of Feb. 9, 1926 to C. F. Wieland and R. H. Hubbell; 1736915 of Nov. 26, 1929 to Robert Illemann and R. A. Whitson; 1862837 of Jun. 14, 1932 to C. F. Wieland and R. H. Hubbell; 1939521 of Dec. 12, 1933 to Georg Reichenbecher and Ernst Grube; **Can. Pat.** 361915 of Nov. 17, 1936 to Standard Oil Co.; **Brit. Pat.** 367387 of Nov. 13, 1930 to Wilhelm Kunst; **Ger. Pat.** 591044 of Jan. 16, 1934 to Vereinigte Stahlwerke, A.-G.

42 [p. 538] **U. S. Pats.** 1792855 of Feb. 17, 1931 to D. F. McRae; 2024602 of Dec. 17, 1935 to R. G. Roberts; **Brit. Pat.** 435325 of Sep. 19, 1935 to S. P. Miller and L. T. Sutherland.

43 [p. 538] **U. S. Pat.** 2030515 of Feb. 11, 1936 to W. A. Kraner.

44 [p. 538] **U. S. Pats.** 1948007 of Feb. 20, 1934 to J. F. Putnam; 1984649 of Dec. 18, 1934 to C. S. McDonald, J. F. Putnam and R. M. McHale; 1988628 of Jan. 22, 1935 to C. S. McDonald, J. F. Putnam and R. M. McHale; **Can. Pat.** 321095 of Mar. 22, 1932 to Standard Oil Co. of Cal.; 363326 and 363327 of Jan. 12, 1937 to Barrett Co.; 365242 of Apr. 6, 1937 to Inertol Co., Inc.; **Brit. Pat.** 350601 of Mar. 20, 1930 to South Durham Steel & Iron Co., Ltd. and David Ross.

45 [p. 538] **U. S. Pat.** 1667201 of Apr. 24, 1928 to Dozier Finley; **Can. Pat.** 247163 of Feb. 24, 1925 to Paraffine Cos., Inc.

46 [p. 538] "Protection of Underground Pipe from Corrosion," by E. O. Slater, *J. Ind. Eng. Chem.*, **21**, 19 (1929); **U. S. Pats.** 131272 of Sep. 10, 1872 to C. W. Hebard; 522946 of Jul. 10, 1894 to Jordan Shultis and D. J. Shultis; 542701 of Jul. 16, 1895 to E. F. Badgley; 566629 of Aug. 25, 1896 to R. D. Smillie; 611208 of Sep. 20, 1898 to V. D. Rood and H. R. Rood; 659437 of Oct. 9, 1900 to A. S. Dixon; 868339 of Oct. 15, 1907 to Edward Gold; 1151096 of Aug. 24, 1915 to J. R. French; 1375979 of Apr. 26, 1921 to R. G. Taber; 1420911 of Jun. 27, 1922 to C. W. Davis and W. S. Brown; 1499774 of Jul. 1, 1924 to F. A. Headson; 1499781 of Jul. 1, 1924 to F. L. Pilliod; 1500769 of Jul. 8, 1924 to L. S. Rosener and F. I. Doane; 1572258 of Feb. 9, 1926 to C. F. Wieland and Emile Jansen; 1622427 of Mar. 29 1927 to C. S. Evans; 1622432 of Mar. 29, 1927 to Dozier Finley; 1625983 of Apr. 26, 1927 to H. F. Carter; 1668265 of May 1, 1928 to H. S. Christopher; 1669555 of May 15, 1928 to Dozier Finley; 1686929 of Oct. 9, 1928 to L. S. Rosener; 1735136 of Nov. 12, 1929 to P. D. Shoenberger; 1742775 of Jan. 7, 1930 to P. D. Mallay; Reissue 17675 of May 27, 1930 to H. S. Christopher; 1777089 of Sep. 30, 1930 to Emile Jansen; 1777095 of Sep. 30, 1930 to Herman Kramer; 1796691 of Mar. 17, 1931 to Emile Jansen; 1807869 of Jun. 2, 1931 to Perry Okey; 1814320 of Jul. 14, 1931 to H. B. Lynch; 1854395 of Apr. 19, 1932 to J. D. Cummings; 1876651 of Sep. 13, 1932 to Dozier Finley; 1876760 of Sep. 13, 1932 to L. S. Rosener; 1880770 of Oct. 4, 1932 to K. H. Burton and Herman Kramer; 1880771 of Oct. 4, 1932 to K. H. Burton and Herman Kramer; 1883401 of Oct. 18, 1932 to E. L. Rolfs and C. W. Fuller; (J.M.Co.); 1986696 of Jan. 1, 1935 to L. H. Wilson; 1998908 of Apr. 23, 1935 to L. S. Rosener and Emil Birk; 1998909, 1998910 and 1998911 of Apr. 23, 1935 to L. S. Rosener; 2034755 of Mar. 24, 1936 to C. W. Fuller; 2037043 of Apr. 14, 1936 to W. R. Postlewaite; 2040876 of May 19, 1936 to W. R. Postlewaite; 2044456 of Jun. 16, 1936 to F. W. Yeager; 2044778 of Jun. 23, 1936 to D. N. Halstead; 2051634 of Aug. 18, 1936 to J. S. Carroll and D. N. Halstead; 2069539 of Feb. 2, 1937 to R. G. Roberts; 2070925 of Feb. 16, 1937 to E. L. Rolfs and C. W. Fuller; **Can. Pat.** 265919 of Nov. 16, 1926 to Paraffine Cos., Inc.; 350700 of Jun. 4, 1935 to Barrett Co.

47 [p. 538] "Protective Coating for Steel Pipe," by L. M. Klauber, *Gas Age Record*, **48**, 478 and 514 (1921); **Brit. Pat.** 375553 of Jun. 26, 1931 to British Mannesmann Tube Co., Ltd.

48 [p. 538] **U. S. Pat.** 2024602 of Dec. 17, 1935 to R. G. Roberts; **Can. Pat.** 314271 of Aug. 11, 1931 to Standard Oil Co. of Cal.; **Brit. Pats.** 354803, 354844 and 354845 of Mar. 17, 1930 to Standard Oil Co. of Cal.; **Ger. Pat.** Appl. St–552.30 of Mar. 18, 1930 to Standard Oil Co. of Cal.

49 [p. 539] **U. S. Pat.** 1876665 of Sep. 13, 1932 to W. R. Greig.

50 [p. 539] **Ger. Pat.** Appl. T-43282 of Nov. 20, 1933 to Adele Thielmann.

51 [p. 539] "Cellophane and Kodapak for Pipe-Line Protection," by Lee Holtz, *Western Gas*, **11**, No. 10, 22 and 56 (1935); **U. S. Pat.** 2054769 of Sep. 15, 1936 to Lee Holtz.

52 [p. 539] **Can. Pat.** 340403 of Mar. 27, 1934 to Cement Wrapped Pipe Co., Ltd.

53 [p. 539] **U. S. Pat.** 2047778 of Jul. 14, 1936 to H. P. Hayden.

54 [p. 539] "Bituminous Joint Compounds for Sewer Pipe," by J. S. Crandell, *Proc. A.S.T.M.*, **34**, Part II, 581 (1934); **U. S. Pats.** 356411 of Jan. 18, 1887 to Terrence Sweeney; 1240287 of Sep. 18, 1917 to E. C. Woodward.

55 [p. 541] "Teerprodukte in der elektrotechnischen Isoliermittelindustrie," by W. Ritter, *Teer*, **6**, 47 (1923); "Herstellung von elektrischen Isoliermaterialien, ein wichitges

Verwertungsfeld für Produkte der Teerindustrie," by Dr. Stade, *Teer*, **25**, 431 (1927); "Ueber die Anwendung von Bitumen in der Kabeltechnik," by E. Da Fano, *Bitumen*, **5**, 93 (1935).

56 [p. 541] **U. S. Pats.** 11096 of Jun. 13, 1854 to Thomas, Earl of Dundonald; 58615 of Oct. 9, 1866 to A. G. Day; 83031 of Oct. 13, 1868 to S. C. Bishop; Reissues 6707 and 6708 of Oct. 19, 1875 to A. G. Day; 286442 of Oct. 9, 1883 to M. Hughes; 315633 of Apr. 14, 1885 to H. Kellogg; 318233 of May 19, 1885 to J. L. Clark; 319079 of Jun. 2, 1885 to J. W. Ellis; 335495 of Feb. 2, 1886 to J. B. Williams; 375436 of Dec. 27, 1867 to S. M. Allen; 452760 and 452765 of May 19, 1891 to Frederick Salathé; 522312 of Jul. 3, 1894 to A. A. Blandy; 580139 of Apr. 6, 1897 to William Morison; 645331 of Mar. 13, 1900 to W. Prampolini; 1935323 of Nov. 14, 1933 to A. R. Kemp and J. H. Ingamnson; **Brit. Pat.** 380693 of Jan. 25, 1932 to Adele Thielmann.

57 [p. 541] **U. S. Pats.** 210407 and 210409 of Dec. 3, 1878 to A. G. Day.

58 [p. 541] **U. S. Pats.** 346224 of Jul. 27, 1886 to T. C. Roche; 359825 of Mar. 22, 1887 to C. M. Thompson.

59 [p. 541] **U. S. Pats.** 34309 of Feb. 4, 1862 to T. J. Mayall; 113588 of Apr. 11, 1871 to J. H. Smyser; 320921 of Jun. 30, 1885 to R. S. Ferguson, W. Schumacher and W. Tubman; 2060856 of Nov. 17, 1936 to J. M. De Bell.

60 [p. 541] **U. S. Pat.** 339787 of Apr. 13, 1886 to E. D. Kendall.

61 [p. 541] **U. S. Pat.** 391927 of Oct. 30, 1888 to J. A. Titzel.

62 [p. 541] **Brit. Pat.** 309851 of Dec. 15, 1927 to W. E. Beatty.

63 [p. 541] **Brit. Pat.** 353311 of Sep. 15, 1930 to Max Deseniss and Andreas Nielsen; **French Pat.** 754119 of Nov. 2, 1933 to Soc. J. R. Geigy, S. A.

64 [p. 541] **U. S. Pat.** 1787629 of Jan. 6, 1931 to J. A. Kennedy.

65 [p. 541] **Ger. Pats.** 454831 of Apr. 9, 1925 to Hans Friedländer; 506883 of Sep. 26, 1925 to Hans Friedländer.

66 [p. 541] **U. S. Pats.** 1921292 and 1921293 of Aug. 8, 1933 to M. T. Harvey.

67 [p. 541] **U. S. Pat.** 517452 of Apr. 3, 1894 to Adolf Gentzsch; **Brit. Pats.** of 1893 (Sep. 7), 16851 to Adolf Gentzsch; of 1901 (Sep. 14), 18417 to Robert Abrey.

68 [p. 541] **Brit. Pat.** of 1885 (Feb. 12), 1958 to R. Punshon and S. H. Beckles; **Ger. Pats.** 9620 of Jun. 18, 1879 to Ludwig Dankwerth and Friedrich Sanders; 66892 of Feb. 2, 1892 to John Robson; 79110 of Sep. 6, 1893 to Adolf Gentzsch.

69 [p. 541] **Ger. Pats.** 205770 of Aug. 31, 1907 to Joseph Michael; 226519 of Aug. 16, 1908 to Eduard Dyckerhoff; 226520 of Feb. 15, 1910 to Eduard Dyckerhoff.

70 [p. 541] "Use of Asphalt-Vaseline Mixtures as Fillers in High-Voltage Work," by J. Göbel, *Chem. Weekblad*, **30**, 562 (1933).

71 [p. 542] "Insulative Properties of Solid Dielectrics," by H. L. Curtis, Scientific Paper 234, Bureau of Standards, Wash., D. C. (1915).

72 [p. 542] "Effect of Moisture on Electrical Properties of Insulating Waxes, Resins and Bitumens," by J. A. Lee and H. H. Lowry, *J. Ind. Eng. Chem.*, **19**, 302 (1927).

73 [p. 542] **U. S. Pat.** 610626 of Sep. 13, 1898 to P. H. Hensen; **Brit. Pat.** 291858 of Jun. 21, 1928 to D. Anderson & Son, Ltd. and R. O. Child.

74 [p. 542] **Brit. Pats.** 156527 of Jan. 5, 1921 to Siemens-Schuckert-Werke, G.m.b.H.; 157119 and 157120 of Jan. 8, 1921 to Siemens-Schuckert-Werke, G.m.b.H.; 315280 of Jul. 10, 1928 to British Celanese, Ltd.; 397405 of Aug. 24, 1933 to British Thomson-Houston Co., Ltd.; **Ger. Pats.** 51554 of Jul. 7, 1890 to T. F. Craddock and John Thom; 227208 of Jun. 20, 1909 to Wilhelm Winter and Ernst Winkler.

75 [p. 542] **U. S. Pat.** 1865629 of Jul. 5, 1932 to E. R. Hanson and M. E. Delaney.

76 [p. 542] **Brit. Pat.** 380776 of Sep. 22, 1932 to Felten & Guilleaume Carlswerke, A.-G.

77 [p. 542] **Ger. Pat.** 587044 of Oct. 28, 1933 to Jaroslaw's Erste Glimmerwaren-Fabrik.

78 [p. 542] **U. S. Pat.** 1935885 of Nov. 21, 1933 to Alexander Meissner.

79 [p. 544] "Service Requirements of Insulating Varnishes," by R. H. Arnold and L. E. Frost, *J. Ind. Eng. Chem.*, **25**, 133 (1933).

80 [p. 544] **U. S. Pat.** 1880050 of Sep. 27, 1932 to M. O. Schur.

81 [p. 544] **Brit. Pat.** 370389 of Dec. 27, 1929 to H. J. Cameron.

82 [p. 544] **U. S. Pat.** 1915311 of Jun. 27, 1933 to G. F. Hadley.

83 [p. 546] **U. S. Pats.** 2012961 of Sep. 3, 1935 to S. P. Miller; 2012968, 2012969 and

2012970 of Sep. 3, 1935 to S. P. Miller; 2042676, 2042677 and 2042678 of Jun. 2, 1936 to S. P. Miller; 2058846 of Oct. 27, 1936 to Herbert Waterman; **Can. Pats.** 350059, 350060 and 350061 of May 7, 1935 to Barrett Co.; 359403 of Jul. 28, 1936 to Barrett Co.

84 [p. 546] "Lignostone; the New Wood Product," by Leopold Vorreiter, *Forstwiss. Cent.*, **56**, 533 (1934); **U. S. Pats.** 441870 of Dec. 2, 1890 to E. T. Greenfield and J. Nagel; 1383749 of Jul. 5, 1921 to R. G. Osborne; 1937417 of Nov. 28, 1933 to E. C. Wallace.

85 [p. 546] **U. S. Pats.** 58484 of Oct. 2, 1866 to John Rouse; 1176284 of Mar. 21, 1916 to C. E. Fuller; 1220292 and 1220293 of Mar. 27, 1917 to J. W. Tucker; 1229439 of Jun. 12, 1917 to C. E. Fuller; 1261114 of Apr. 2, 1918 to C. E. Fuller; 1920627 of Aug. 1, 1933 to W. O. Brassert; **Can. Pat.** 340985 of Apr. 17, 1934 to Warren Brothers Co.; **Brit. Pats.** 402845 of Dec. 14, 1933 to William Adams and E. M. Adams; 426162 of Jun. 4, 1934 to Maschinenfabrik Kommick, G.m.b.H.; **Ger. Pats.** 24489 of Oct. 21, 1882 to Richard Michelet and Louis Tescher; Appl. K-313.30 of Sep. 3, 1930 to Ivan Kardos; "New Ceramic Table Tops," by S. M. Phelps and E. E. Marbaker, *J. Ind. Eng. Chem.*, **29**, 541 (1937).

86 [p. 546] **U. S. Pats.** 865606 of Sep. 10, 1907 to C. L. Norton; 1516045 of Nov. 18, 1924 to Heinrich Kollbrunner; 1982314 of Nov. 27, 1934 to J. W. Ledeboer.

87 [p. 546] **Brit. Pats.** 409760 of May 10, 1934 to N. V. de Bataafsche Petroleum Maatschappij; 410479 of May 10, 1934 to N. V. de Bataafsche Petroleum Maatschappij.

88 [p. 546] **U. S. Pat.** 1203577 of Nov. 7, 1916 to H. W. Broady.

89 [p. 546] **U. S. Pats.** 1137030 of Apr. 27, 1915 to E. C. Smith; 1143181 of Jun. 15, 1915 to M. A. Foos; 1196225 of Aug. 29, 1916 to M. A. Foos; 1215774 of Feb. 13, 1917 to E. F. Callender; 1225306 of May 8, 1917 to R. C. Benner; 1328027 of Jan. 13, 1920 to Sakizo Yai; 1345057 of Jun. 29, 1920 to G. O. Bassett; 1431859 of Oct. 10, 1922 to Sakizo Yai; 1500477 of Jul. 8, 1924 to H. E. Warnecke; 1501084 of Jul. 15, 1924 to F. H. Wright and F. A. Keller.

90 [p. 547] **Brit. Pat.** 155226 of Aug. 10, 1920 to Barrett Co.

91 [p. 547] "The Use of Petroleum Coke in the Aluminum Industry," by E. H. Lewis, *Atlantic Connecting Rod*, **15**, 19 (1922).

92 [p. 547] **U. S. Pat.** 1283229 of Oct. 29, 1918 to R. A. Lee.

93 [p. 547] **U. S. Pat.** 452765 of May 19, 1891 to Frederick Salathé.

94 [p. 547] **U. S. Pat.** 1961306 of Jun. 5, 1934 to W. S. Smith, H. J. Garnett, J. N. Dean, H. C. Channon, William Gardner and H. F. Wilson; **Brit. Pat.** 322208 of Aug. 25, 1928 to W. S. Smith, H. J. Garnett, J. N. Dean, H. C. Channon, William Gardner and H. F. Wilson.

95 [p. 548] **U. S. Pat.** 140366 of Jul. 1, 1873 to W. H. Haight and T. S. Gladding; **Brit. Pat.** of 1603 (Jun. 25), 1603 to William Kirrage; **French Pat.** 434052 of Sep. 8, 1911 to Chemische Fabrik Lindenhof, C. Weyl & Co.

96 [p. 548] **Brit. Pat.** 309421 of Jan. 4, 1928 to I. G. Farbenindustrie, A.-G.

97 [p. 548] **U. S. Pat.** 1688041 of Oct. 16, 1928 to C. N. Forrest and J. S. Miller, Jr.; **Brit. Pat.** of 1897 (May 24), 12829 to H. C. P. Graves; **Ger. Pats.** 119635 and 119637 of Apr. 1, 1901 to Dr. Zühl and Eisemann.

98 [p. 548] "Crude Rubber and Compounding Ingredients," by H. C. Pearson, 3rd Edition, India Rubber Publishing Co., New York (1918); "Mineral Rubber," by A. Dubosc and J. Wavelet, *J. Soc. Chem. Ind.*, **38**, 954A (1919); "Emarex—its Origin and Uses," by Charles Muller, *Doherty News*, **5**, 15 (1920); "Mineral Rubber," by C. O. North, *India Rubber World*, **65**, 191 (1921); *Chem. Met. Eng.*, **26**, 253 (1922); "Rubber Softeners and their Influence on Aging," by W. N. Burbridge, *Trans. Inst. Rubber Ind.*, **2**, 256 (1926); "Chemistry of India Rubber," by Lothar Weber, 4th Edition, Chas. Griffin & Co., London (1926); "Verwendung von Bitumen in der Kautschuktechnik," by E. Adel, *Bitumen*, **2**, 50 (1932); "Bitumen und Kautschuk," by Leo Eck, *Teer u. Bitumen*, **35**, 606 (1929); "Rubber as an Electrical Insulating Material," by Th. Wasserburger, *Kautschuk*, **10**, 66 and 91 (1934); "The Testing of Bituminous Mixtures," by D. C. Broome, Edward Arnold & Co., London (1934); "Die Verwendung von Bitumen als 'Mineral Rubber' in der Gummiindustrie," by F. C. van Heurn, *Bitumen*, **5**, 121 and 149 (1935); "Rubber, Bitumens and Road Surfaces, by Harry Barron, *India Rubber J.*, **90**, No. 17a, 23 (1935); "The Rational Compounding of Ebonite Stocks," by B. L. Davies, *India Rubber J.*, **89**, 427 (1935); "Gummi-Bitumen-Mischungen," by Th. Temme, *Teer u. Bitumen*, **34**, 213 (1936); "Bitumen as 'Mineral-Rubber' in Rubber Technology," by Walter Becker, *Kautschuk*, **12**, 131 (1936).

99 [p. 548] **U. S. Pats.** 734483 of Jul. 21, 1903 to S. R. Whitall; 1104140 of Jul. 21, 1914 to J. C. Ross; 1265108 of May 7, 1918 to Paul Pickl.

100 [p. 548] **Brit. Pats.** of 1861 (Jun. 19), 1580 to J. F. Williams; of 1863 (Jun. 25), 1603 to William Kirrage.

101 [p. 549] **U. S. Pats.** 210405, 210406, 210407, 210408, 210409, 210410 and 210411 of Dec. 3, 1878 to A. G. Day.

102 [p. 549] **U. S. Pats.** 1282505 of Oct. 22, 1918 to J. M. Weiss; 1334060 and 1334061 of Mar. 16, 1920 to J. M. Weiss.

103 [p. 549] **U. S. Pats.** 393838 of Dec. 4, 1888 to W. Kiel; 420648 of Feb. 4, 1890 to J. B. Williams; 438313 of Oct. 14, 1890 to O. A. Enholm; **Can. Pat.** 276640 of Dec. 27, 1927 to T. V. Binmore; **Brit. Pats.** of 1891 (Jun. 25), 10822 to C. N. Jackson; 419801 of Feb. 15, 1933 to Chemieprodukte, A.-G.; **Ger. Pat.** 79110 of Sep. 5, 1893 to Adolf Gentzsch.

104 [p. 549] **U. S. Pats.** 310957 of Jan. 20, 1885 to Alexander Muirhead; 1315109 and 1315110 of Sep. 2, 1919 to G. M. Garlick; 1880036 of Sep. 27, 1932 to E. W. Lovering; **French Pat.** 776548 of Jan. 28, 1935 to Joseph Omnes.

105 [p. 549] **U. S. Pats.** 1285463 of Nov. 19, 1918 to E. R. Talley; 1379149 and 1379150 of May 24, 1921 to J. C. Wichmann; **Brit. Pat.** 382557 of Apr. 19, 1932 to Sri Krishna.

106 [p. 549] **U. S. Pats.** 1371016 of Mar. 8, 1921 to C. H. Bennett, J. F. Palmer and F. V. Wedlock; 1435359 and 1435360 of Nov. 14, 1922 to J. C. Wichmann; **Brit. Pats.** 201006 of Jun. 23, 1922 to J. E. Claxton; 263028 of May 29, 1926 to Joseph Campbell; 319904 of Sep. 4, 1928 to Hewitt, Ltd.

107 [p. 549] **French Pat.** 396766 of Nov. 17, 1908 to A. Kraenzlé.

108 [p. 549] **U. S. Pat.** 1395396 of Nov. 1, 1921 to A. C. de Caudemberg; **Can. Pat.** 68779 of Sep. 20, 1900 to C. A. C. de Caudemberg; **Ger. Pat.** 116126 of Dec. 10, 1899 to C. A. C. de Caudemberg.

109 [p. 549] **U. S. Pat.** 506745 of Oct. 17, 1893 to T. C. Stanley; **Brit. Pat.** of 1896 (Aug. 20), 18485 to William Morison.

110 [p. 549] **U. S. Pat.** 74606 of Feb. 18, 1868 to H. K. Schanck; **Brit. Pat.** of 1899 (Dec. 20), 25216 to G. E. Heyl-Dia.

111 [p. 549] **U. S. Pats.** 68106 of Aug. 27, 1867 to J. D. Numan, J. T. Wilkinson and E. W. Cook; 108666 of Oct. 8, 1870 to J. T. Wilkinson.

112 [p. 549] **U. S. Pat.** 1884240 of Oct. 25, 1932 to E. O. Rhodes and F. D. Hager.

113 [p. 549] **U. S. Pats.** 1220401 of Mar. 27, 1917 to A. C. de Caudemberg; 1743433 of Jan. 14, 1930 to A. B. Cowdery; **Ger. Pat.** 399366 of Jan. 8, 1922 to Domingo Sanguinetti.

114 [p. 549] **Ger. Pat.** 49162 of Mar. 8, 1889 to S. A. A. Blandy; **French Pat.** 347943 of Nov. 18, 1904 to H. C. Bouet.

115 [p. 549] **U. S. Pat.** 2009712 of Jul. 30, 1935 to P. K. Frolich.

116 [p. 549] **U. S. Pat.** 2054114 of Sep. 15, 1936 to Allen Abrams and C. L. Wagner; **Can. Pats.** 285960 and 285983 of Dec. 25, 1928 to H. V. Dunham.

117 [p. 549] **U. S. Pat.** 875298 of Dec. 31, 1907 to E. W. Strain.

118 [p. 549] **U. S. Pat.** 375436 of Dec. 27, 1887 to S. M. Allen; **Brit. Pat.** 447416 of Nov. 16, 1934 to J. Lewis; **Ger. Pat.** 121316 of Aug. 10, 1899 to L. H. Lang.

119 [p. 549] **Brit. Pat.** of 1885 (Feb. 12), 1958 to R. Punshon and S. H. Beckles.

120 [p. 549] **U. S. Pat.** 837193 of Nov. 27, 1906 to C. A. C. de Caudemberg.

121 [p. 549] **U. S. Pat.** 977423 of Nov. 29, 1910 to Henry Mielck and F. C. von Heydebrand.

122 [p. 549] **U. S. Pat.** 286442 of Oct. 9, 1883 to Michael Hughes.

123 [p. 549] **Ger. Pat.** 168048 of Feb. 24, 1904 to Pierre Lacollonge.

124 [p. 549] **Brit. Pat.** 164392 of Mar. 24, 1920 to F. L. Schauerman.

125 [p. 549] **U. S. Pat.** 1875294 of Aug. 30, 1932 to Charles Cholerton.

126 [p. 549] **U. S. Pat.** 1217157 of Feb. 27, 1917 to M. F. Coughlin.

127 [p. 549] **U. S. Pat.** 610626 of Sep. 13, 1898 to P. H. Hansen; **Brit. Pat.** of 1897 (Jun. 11), 14272 to P. H. Hansen.

128 [p. 549] **Ger. Pat.** 113638 of Aug. 30, 1898 to G. Verbeeckmoes.

129 [p. 550] **Ger. Pat.** 219305 of Apr. 12, 1907 to Curt Heinrichsdorff and Rudolf Zimpel.

130 [p. 550] **U. S. Pat.** 1709242 of Apr. 16, 1929 to H. A. Winkelmann.

131 [p. 550] "Synthetic Resins and their Plastics," by Carleton Ellis, The Chemical Catalog Co., Inc., New York (1923); "Plastics and Molded Electrical Insulation," by Emile Hemming, The Chemical Catalog Co., Inc., New York (1923); "The Chemistry of Synthetic Resins," by Carleton Ellis, Reinhold Publishing Corp., New York (1935).

132 [p. 550] **Can. Pat.** 318381 of Dec. 29, 1931 to G. H. Killer.

133 [p. 550] **U. S. Pat.** 1939492 of Dec. 12, 1933 to H. C. Fischer and C. L. Keller.

134 [p. 550] **U. S. Pats.** 259271 of Jun. 6, 1882 to J. A. Fleming; 517452 of Apr. 3, 1894 to Adolf Gentzsch.

135 [p. 550] **U. S. Pat.** 86355 of Feb. 2, 1869 to J. W. Brown; **Austrian Pat.** 55651 of Nov. 1, 1913 to H. P. Rasmussen.

136 [p. 550] **U. S. Pat.** 1346397 of Jul. 13, 1920 to J. F. Cavanagh.

137 [p. 550] **U. S. Pat.** 2040671 of May 12, 1936 to L. T. Richardson.

138 [p. 550] **U. S. Pat.** 2057456 of Oct. 13, 1936 to R. B. Stringfield.

139 [p. 550] **Can. Pat.** 323860 of Jul. 5, 1932 to Barrett Co.

140 [p. 550] **U. S. Pat.** 1711544 of May 7, 1929 to Junjiro Sato and Tsunaji Sato.

141 [p. 550] **Brit. Pat.** 397405 of Mar. 18, 1933 to British Thomson-Houston Co., Ltd.

142 [p. 550] **U. S. Pat.** 1944464 of Jan. 23, 1934 to L. T. Richardson.

143 [p. 550] **U. S. Pat.** 1966094 of Jul. 10, 1934 to C. A. Herbst; 2054815 of Sep. 22, 1936 to C. A. Herbst.

144 [p. 550] **U. S. Pat.** 1825620 of Sep. 29, 1931 to R. E. Coleman.

145 [p. 550] **U. S. Pat.** 1732478 of Oct. 22, 1929 to Frank Kurath.

146 [p. 550] **U. S. Pat.** 1596760 of Aug. 17, 1926 to C. A. Nash; **Ger. Pats.** 145250 of Oct. 19, 1903 to Victor de Karavodine; 215682 of Oct. 28, 1909 to Ludwig Grote; **Austrian Pat.** 58735 of Nov. 15, 1912 to Albin Baer.

147 [p. 550] **Brit. Pat.** 366304 of Feb. 20, 1930 to Nicolaas van der Willik; **French Pat.** 775906 of Jan. 12, 1935 to Urbain Chandeysson.

148 [p. 551] **U. S. Pat.** 748019 of Dec. 29, 1903 to Carl Rubitschung.

149 [p. 551] **Ger. Pats.** 5646 of Oct. 23, 1878 to J. S. Kahlbetzer; 5678 of Nov. 10, 1878 to Zadig & Neuberg; 9307 of Aug. 30, 1879 to A. H. Elliot and A. Walcker.

150 [p. 551] **U. S. Pat.** 1935794 of Nov. 21, 1933 to H. D. Geyer; **Ger. Pats.** 92314 of May 16, 1896 to Anton Pieper; 103055 of Jan. 29, 1898 to Anton Pieper; 127165 of Nov. 15, 1900 to Schliemann & Co.

151 [p. 551] "Plastic Molding—An Introduction to the Materials, Equipment and Methods Used in the Fabrication of Plastic Products," by L. F. Rahm, McGraw-Hill Book Co., Inc., New York (1933).

152 [p. 551] **U. S. Pats.** 1785457 of Dec. 16, 1930 to A. J. Pfohl; 1944464 of Jan. 23, 1934 to L. T. Richardson; 1952038 of Mar. 20, 1934 to A. C. Fischer; 1977515 of Oct. 16, 1934 to H. B. Klippel; 1993343 of Mar. 5, 1935 to E. V. Harlow; Reissue 19553 of Apr. 30, 1935 to A. J. Pfohl.

153 [p. 551] **U. S. Pat.** 724079 of Mar. 31, 1903 to Hans Biehm.

154 [p. 551] **U. S. Pat.** 1593376 of Jul. 20, 1926 to T. A. Wry.

155 [p. 551] **U. S. Pat.** 1643059 of Sep. 20, 1927 to F. C. J. de Both; **Ger. Pat.** Appl. B–152793 of Oct. 31, 1931 to Bosari & Co.

156 [p. 551] **Brit. Pats.** 212106 of Mar. 3, 1923 to Building Accessories & Flooring Co., Ltd., Harold Goodwin and Harry Smith; 212188 of Aug. 21, 1923 to Building Accessories & Flooring Co., Ltd., Harold Goodwin and Harry Smith.

157 [p. 551] **U. S. Pat.** 1869141 of Jul. 26, 1932 to H. D. Geyer.

158 [p. 552] **U. S. Pats.** 1227861 of May 29, 1917 to W. P. Waugh; 1243020 of Oct. 16, 1917 to W. P. Waugh; **Ger. Pat.** 71478 of Apr. 6, 1893 to D. H. W. Schultz & Sohn.

159 [p. 552] **U. S. Pats.** 1447347 of Mar. 6, 1923 to Lester Kirschbraun; 1452555 of Apr. 24, 1923 to F. J. Gleason; 1466907 of Sep. 4, 1923 to Lester Kirschbraun; 1851036 of Mar. 29, 1932 to T. F. Brackett; **Can. Pats.** 325195 of Aug. 16, 1932 to Timken-Detroit Axle Co.; 328810 of Dec. 27, 1932 to American Brake Materials Corp.; **Brit. Pat.** 374390 of Jan. 26, 1932 to W. S. Skerrett.

160 [p. 552] **U. S. Pats.** 452182 of May 12, 1891 to F. C. Goodall; 841875 of Jan. 22, 1907

to Eugene Kolbassieff; 1162450 of Nov. 30, 1915 to C. P. Byrnes; 1218444 of Oct. 15, 1918 to H. R. Wardell; 1285888 of Nov. 26, 1918 to J. E. Allen; **Can. Pats.** 34591 of Jun. 28, 1890 to Patent Cork Pavement Co.; 35971 of Feb. 9, 1891 to F. C. Goodall; 37001 of Jul. 16, 1891 to R. F. Flynn and John Guthrie; **Brit. Pats.** of 1888 (May 30), 7933 to O. Imray; of 1890 (May 6), 7028 to R. F. Flynn; of 1906 (Apr. 2), 7853 to C. M. C. Hughes, T. H. Quinlan and R. Middleton; of 1906 (Sep. 27), 21437 to Cork Asphalt, Ltd. and H. M. Clifford; **Ger. Pats.** 48154 of Aug. 15, 1888 to P. Martiny and A. Grupe; 285368 of Apr. 2, 1914 to E. Müller and E. Eichert.

161 [p. 552] **U. S. Pat.** 941585 of Nov. 30, 1909 to V. A. Noodt.

162 [p. 553] "Teer, Bitumen und Kork," by Walter Obst, *Teer u. Bitumen*, **34**, 354 (1936); **U. S. Pats.** 395438 of Jan. 1, 1889 to Carl Grünzweig; 674969 of May 28, 1901 to Otto Horstmann; 778529 of Dec. 27, 1904 to F. H. Bruening; 979310 of Dec. 20, 1910 to W. C. Kammerer; 2070330 of Feb. 9, 1937 to F. L. Chase; **Brit. Pat.** of 1900 (Aug. 16), 14722 to Otto Horstmann & Co.; **Ger. Pats.** 122803 of Sep. 29, 1900 to Nafzger & Rau; 128231 of Nov. 11, 1899 to A. Haacke; 267733 of Dec. 8, 1906 to Grünzweig & Hartmann; 277638 of May 1, 1912 to Grünzweig & Hartmann; **Swiss Pat.** 22072 of Aug. 20, 1900 to Otto Horstmann & Co.

163 [p. 553] **U. S. Pats.** 1302810 of May 6, 1919 to Lester Kirschbraun; 1660916 of Feb. 28, 1928 to M. L. Billard; 1884740 of Oct. 25, 1932 to Lester Kirschbraun; **Brit. Pats.** of 1900 (Mar. 17), 5108 to A. Haacke; 319648 of Aug. 22, 1928 to Bowran & Co., Ltd. and J. W. Craggs; **Ger. Pat.** 68532 of Jul. 1, 1891 to Grünzweig & Hartmann.

164 [p. 553] **Can. Pat.** 309672 of Mar. 24, 1931 to Armstrong Cork Co.

165 [p. 553] **U. S. Pats.** 122925 of Jan. 23, 1872 to W. H. Towers; 391265 of Aug. 25, 1891 to S. H. Howland; 636657 of Nov. 7, 1899 to Franz Gatzsche; 808224 of Dec. 26, 1905 to Andrew Thoma; 808227 of Dec. 26, 1905 to W. B. Arnold; 832002 of Sep. 25, 1906 to Andrew Thoma; 855868 of Jun. 4, 1907 to Andrew Thoma; 861555 of Jul. 30, 1907 to Andrew Thoma; 945294 of Jan. 4, 1910 to Andrew Thoma; 1004070 of Sep. 26, 1911 to Angelo Perri; 1032312 of Jul. 9, 1912 to Andrew Thoma; 1036931 of Aug. 27, 1912 to Andrew Thoma; 1115988 of Nov. 3, 1914 to Andrew Thoma; 1118161 of Nov. 24, 1914 to Andrew Thoma; 1121054 of Dec. 15, 1914 to Andrew Thoma; 1121688 of Dec. 22, 1914 to Andrew Thoma; 1121689 of Dec. 22, 1914 to Andrew Thoma; 1134931 of Apr. 6, 1915 to Andrew Thoma; 1136456, 1136457 and 1136458 of Apr. 20, 1915 to F. E. Woodward; 1137679 of Apr. 27, 1915 to Andrew Thoma; 1138909 of May 11, 1915 to Andrew Thoma; 1224153 of May 1, 1917 to Otto Eisenschiml; 1227502 of May 22, 1917 to Andrew Thoma; 1258272 of Mar. 5, 1918 to H. S. Tirrell; 1796670 of Mar. 17, 1931 to Andrew Thoma; 1920771 of Aug. 1, 1933 to Andrew Thoma; 2025432 of Dec. 24, 1935 to H. H. Beckwith; 2052579 of Sep. 1, 1936 to J. A. Muir; **Brit. Pat.** 319648 of Aug. 22, 1928 to Bowran & Co., Ltd. and J. W. Craggs.

166 [p. 553] **U. S. Pats.** 1124835 of Jan. 12, 1915 to H. H. Beckwith; 1204844 of Nov. 14, 1916 to Howard Brummett and W. H. Colbath; 1209661 of Dec. 26, 1916 to H. H. Beckwith; 1263187 of Apr. 16, 1918 to W. G. Abbott, Jr.; 1263499 of Apr. 23, 1918 to M. T. Whiting; 1312758 of Aug. 12, 1919 to G. A. Spear; 1316650 of Sep. 23, 1919 to A. J. Ryan; 1473695 of Nov. 13, 1923 to Max Brown; 1801463 of Apr. 21, 1931 to Andrew Thoma; **Brit. Pats.** of 1861 (Feb. 1), 271 to J. J. de Arrieta; of 1888 (Feb. 21), 2543 to H. G. Boston and R. Combe; of 1889 (Aug. 14), 12844 to T. H. Wright; of 1895 (Sep. 2), 16399 to Edward Nelson.

167 [p. 553] **U. S. Pat.** 1284023 of Nov. 5, 1918 to G. R. Wyman and A. E. Currier.

168 [p. 553] **U. S. Pats.** 122925 of Jan. 23, 1872 to W. H. Towers; 636657 of Nov. 7, 1899 to Frank Gatzsche; 1385808 of Jul. 26, 1921 to Herbert Abraham; 1868927 and 1868928 of Jul. 26, 1932 to Andrew Thoma; **Brit. Pat.** 180817 of Jun. 8, 1922 to Herbert Abraham; **Ger. Pat.** 364614 of Mar. 6, 1921 to Herbert Abraham.

169 [p. 553] **U. S. Pats.** 268034 of Nov. 28, 1882 to Murdoch Mackay; 284289 of Sep. 4, 1883 to J. A. Fleming; 319084 of Jun. 2, 1885 to J. A. Fleming; 337472 of Mar. 9, 1886 to S. M. Allen; 361759 of Apr. 26, 1887 to S. H. Gilson; 373334 of Nov. 15, 1887 to C. A. Brown; 393382 of Nov. 27, 1888 to F. A. Meyer; 411124 of Sep. 17, 1889 to F. W. Huestis; 415962 of Nov. 26, 1889 to O. A. Enholm; 429999 of Jun. 10, 1890 to C. A. Catlin; 438311 of Oct. 14, 1890 to O. A. Enholm; 452760 and 452763 of May 19, 1891 to Frederick Salathé; 499354

of Jun. 13, 1893 to J. de S. Brown; 505916 of Oct. 3, 1893 to Joseph Hoffman; 522312 of Jul. 3, 1894 to A. D. Blandy; 571117 of Nov. 10, 1896 to F. R. Hall; 580139 of Apr. 6, 1897 to William Morison; 656700 of Aug. 28, 1900 to L. W. Pullen; 662929 of Dec. 4, 1900 to Willibald Gelinek; 716787 of Dec. 23, 1902 to R. M. Thomson; 724882 of Apr. 7, 1903 to Victor de Karavodine; 838419 of Dec. 11, 1906 to Victor de Karavodine; 869321 of Oct. 29, 1907 to Robert Müller; 907552 of Dec. 22, 1908 to P. Zürn; 910989 of Jan. 26, 1909 to P. J. Cannon, W. H. Young and Clinton Bateholts; 918649 of Apr. 20, 1909 to A. G. Betts; 930524 of Aug. 10, 1909 to W. A. Biles; 939982 of Nov. 16, 1909 to F. J. Conboy; 941585 of Dec. 7, 1909 to V. A. Noodt and G. Goettsche; 977423 of Nov. 29, 1910 to Henry Mielck and F. C. von Heydebrand; 1025268 of May 7, 1912 to Emile Hemming; 1152783 of Sep. 7, 1915 to Heinrich Bosari-Fischer; 1156122 of Oct. 12, 1915 to J. C. Woodley; 1166166 of Dec. 28, 915 to J. C. Woodley; 1197022 of Sep. 5, 1916 to E. G. Gallagher; 1302810 of May 6, 1919 to Lester Kirschbraun; 1369783 of Mar. 1, 1921 to R. N. Chamberlain; 1379366 of May 24, 1921 to R. L. Seabury; 1456565 of May 29, 1923 to R. B. Owen; 1467311 of Sep. 11, 1923 to J. P. Elliott; 1512313 of Oct. 21, 1924 to J. G. Rowe; 1517360 of Dec. 2, 1924 to F. J. Groten, Jr.; 1610765 of Dec. 14, 1926 to E. F. Germain; 1623588 of Apr. 5, 1927 to E. F. Germain; 1655388 of Jan. 3, 1928 to F. J. Groten, Jr.; 1732478 of Oct. 22, 1929 to Frank Kurath; 1785910 of Dec. 23, 1930 to C. P. Nellis and A. H. Flower; 1886269 of Nov. 1, 1932 to R. N. Chamberlain; 1892703 of Jan. 3, 1933 to C. T. G. Lindh; 1979012 of Oct. 30, 1934 to J. H. Reilly; 1984307 of Dec. 11, 1934 to C. L. Keller; 2027483 of Jan. 14, 1936 to C. L. Keller; **Can. Pat.** 298098 of Mar. 11, 1930 to Junjiro Sato and Tsunaji Sato; **Brit. Pats.** of 1860 (Jun. 21), 1503 to John Smith; of 1869 (Feb. 18), 507 to Thomas Forster; of 1875 (Jun. 9), 4267 to Alexander Wilkinson; of 1880 (Jan. 9), 94 to Richard Condy; of 1882 (May 22), 2414 to J. A. Fleming; of 1884 (Apr. 22), 6647 to J. A. Fleming; of 1891 (Jun. 3), 9434 to John Menzies; of 1897 (Apr. 12), 9290 to William Morison; of 1899 (Jul. 10), 14227 to F. A. M. Kaempf; of 1901 (Sep. 14), 18417 50 Robert Abrey; of 1902 (Oct. 4), 21650 to J. K. Field; of 1904 (Mar. 16), 6402 to British Thomson-Houston Co., Ltd.; of 1904 (Sep. 1), 18920 to H. H. Lake; of 1904 (Sep. 2), 18989 to Robert Müller; of 1910 (May 3), 11908 to A. R. Müller; 260505 of May 11, 1926 to R. H. Abrey; 305973 of Nov. 11, 1927 to Edward Thomas; 458305 of Mar. 11, 1935 to E. R. Dillehay; **Ger. Pats.** 119759 of Aug. 18, 1899 to Willibald Gelinek; 167166 of Jan. 12, 1907 to Robert Müller; 219305 of Apr. 12, 1907 to Curt Heinrichsdorff and Rudolf Zimpel; **French Pat.** 333928 of Jul. 4, 1903 to J. K. Field.

170 [p. 553] **U. S. Pats.** 1556703 of Oct. 13, 1925 to A. R. Lukens; 1752917 of Apr. 1, 1930 to A. R. Lukens; **Can. Pat.** 295938 of Dec. 24, 1929 to Canadian Battery Container Corp., Ltd.

171 [p. 553] **U. S. Pat.** 1623588 of Apr. 5, 1927 to E. F. Germain; **Can. Pat.** 262933 of Jul. 27, 1926 to J. G. Rowe.

172 [p. 554] **U. S. Pats.** 18278 of Sep. 29, 1857 to R. T. Havens; 52978 of Mar. 6, 1866 to Andrew Doudes; 63371 of Apr. 2, 1867 to D. W. Denton; 72727 of Dec. 21, 1867 to A. B. Ely; Reissue 2969 of Jun. 9, 1886 to A. B. Ely; 88516 of Mar. 30, 1869 to R. W. Russell; 91576 of Jun. 22, 1869 to H. G. Soules; 98065 of Dec. 21, 1869 to C. B. Hutchins; 118850 of Sep. 12, 1871 to C. L. Fowler; 122962 of Jan. 23, 1872 to C. H. Pond; 210204 of Nov. 26, 1878 to Adolphe Kempenner; 223880 of Jan. 27, 1880 to J. Burbridge, R. C. Thorpe and T. Oakley; 239951 of Apr. 12, 1881 to W. M. Graze; 248324 of Oct. 18, 1881 to H. W. Johns; 268034 of Nov. 28, 1882 to Murdoch Mackey; 300464 of Jun. 17, 1884 to Levi Haas; 319084 of Jun. 2, 1885 to J. A. Fleming; 334974 of Jan. 26, 1886 to A. A. Oliver; 337472 of Mar. 9, 1886 to S. M. Allen; 352445 of Nov. 9, 1886 to J. W. Butler; 372552 of Nov. 1, 1887 to Terrence McSweeney; 393644 of Nov. 27, 1888 to Salomon Heimann; 436733 of Sep. 16, 1890 to J. W. Easton; 461467 of Oct. 20, 1891 to M. O. Farrar and C. C. Howe; 505916 of Oct. 3, 1893 to Joseph Hoffman; 571117 of Nov. 10, 1896 to F. R. Hall; 636657 of Nov. 7, 1899 to Franz Gatzsche; 662929 of Dec. 4, 1900 to Willibald Gelinek; 1265655 of May 7, 1918 to G. A. Henderson; 1270732 of Jun. 25, 1918 to G. A. Henderson; 1282009 of Oct. 15, 1918 to J. F. Collins; 1305125 of May 27, 1919 to J. J. Laing and C. W. Boling; 1340181 of May 18, 1920 to G. L. Oliensis and J. S. Miller, Jr.; 1442002 of Jan. 9, 1923 to Frederick Reisig, Sr.; 1449745 of Mar. 27, 1923 to A. L. Clapp; 1476570 of Dec. 4, 1923 to F. W. Adams; 1556641 of Oct.

13, 1925 to Max Skolnik; 1587462 of Jun. 1, 1926 to F. W. Adams; 1599253 of Sep. 7, 1926 to Max Skolnik; 1732281 of Oct. 22, 1929 to Thomas Robinson; 1751690 of Mar. 25, 1930 to C. R. Faben; 1760713 of May 27, 1930 to G. F. A. Ochs; 1838658 of Dec. 29, 1931 to A. B. Chapin; 1874633 of Aug. 30, 1932 to Junjiro Sato and Shigeru Timikawa; 2010034 of Aug. 6, 1935 to L. T. Richardson; **Can. Pats.** 7314 of Apr. 9, 1877 to C. B. Hutchins; 204941 of Oct. 19, 1920 G. L. Oliensis and J. S. Miller, Jr.; 233543 of Aug. 14, 1923 to A. L. Clapp; **Brit. Pat.** 256640 of Aug. 6, 1926 to Prodorite, Ltd.; **Austrian Pats.** 2002 of May 1, 1900 to Franz Gatzsche; 6443 of Aug. 15, 1901 to Willibald Gelinek.

173 [p. 554] **U. S. Pat.** 1831779 of Nov. 10, 1931 to C. R. MacDonald; **Can. Pat.** 278560 of Mar. 13, 1928 to Flintkote Co.

174 [p. 554] **U. S. Pats.** 1290954 of Jan. 14, 1919 to S. M. Ford; 1977955 of Oct. 23, 1934 to Thomas Robinson; 2025077 of Dec. 24, 1935 to B. A. Stewart; 2072958 of Mar. 9, 1937 to S. P. Miller; **Can. Pat.** 311714 of May 26, 1931 to Lancaster Asphalt, Inc.

175 [p. 554] **U. S. Pats.** 1177267 of Mar. 28, 1916 to R. P. Perry; 1253386 of Jan. 15, 1918 to I. S. Joseph; 1293293 of Feb. 4, 1919 to B. A. Allison.

176 [p. 554] **U. S. Pats.** 1000598 of Aug. 15, 1911 to J. B. Hall; 1417840 of May 30, 1922 to Lester Kirschbraun; 1606427 of Nov. 9, 1926 to Lester Kirschbraun; 1659401 of Feb. 14, 1928 to Lester Kirschbraun.

177 [p. 554] **U. S. Pats.** 1156122 of Oct. 12, 1915 to J. C. Woodley; 1166166 of Dec. 28, 1915 to J. C. Woodley; 1240524 of Sep. 18, 1917 to J. C. Woodley; 1265259 of May 7, 1918 to A. E. Schutte; 1386276 of Aug. 2, 1921 to R. P. Perry; 1498401 of Jun. 17, 1924 to R. P. Perry; 1550648 of Aug. 18, 1925 to R. P. Perry; 1698733 of Jan. 15, 1929 to R. P. Perry; 1710320 of Apr. 23, 1929 to R. P. Perry; 1891476 of Dec. 20, 1932 to C. L. Keller; 1891918 of Dec. 27, 1932 to E. R. Dillehay; **Ger. Pat.** Appl. W-46681 of Jul. 3, 1915 to J. C. Woodley (withdrawn).

178 [p. 554] **U. S. Pats.** 1878625 of Sep. 20, 1932 to W. C. Ferguson; Reissue 19381 of Nov. 27, 1934 to W. C. Ferguson.

179 [p. 554] **U. S. Pats.** 1000598 of Aug. 15, 1911 to J. B. Hall; 1379590 of May 24, 1921 to A. L. Clapp; 1767532 of Jun. 24, 1930 to Lester Kirschbraun; 1781645 of Nov. 11, 1930 to Lester Kirschbraun; 1782855 of Nov. 25, 1930 to H. L. Levin; 1889473 of Nov. 29, 1932 to E. O. Groskopf; **Brit. Pat.** 440443 of Jul. 19, 1934 to H. Stössel; **French Pat.** 734382 of Mar. 30, 1932 to Franz Gassner; **Austrian Pat.** 129775 of Apr. 15, 1932 to Eduard Russwurm.

180 [p. 554] **U. S. Pat.** 1940974 of Dec. 26, 1933 to G. D. Shaver.

181 [p. 554] **U. S. Pats.** 1968851 and 1969519 of Aug. 7, 1934 to O. F. Mottweiler.

182 [p. 554] **U. S. Pat.** 1785910 of Dec. 23, 1930 to C. P. Nellis and A. H. Flower.

183 [p. 554] **U. S. Pat.** 1277322 of Aug. 27, 1918 to W. V. Lander.

184 [p. 554] **U. S. Pat.** 1785457 of Dec. 16, 1930 to A. J. Pfohl; **Can. Pat.** 317879 of Dec. 8, 1931 to Patent & Licensing Corp.

185 [p. 554] **Can. Pat.** 313484 of Jul. 21, 1931 to Flintkote Co.

186 [p. 554] **U. S. Pat.** 1971100 of Aug. 21, 1934 to A. C. Fischer.

187 [p. 554] **U. S. Pat.** 1875867 of Sep. 6, 1932 to S. H. Hartman and H. L. Powers.

188 [p. 554] **U. S. Pats.** 117601 of Aug. 1, 1871 to C. W. Chaffee; 836223 of Nov. 20, 1906 to S. E. Stevenson; 1051769 of Jan. 28, 1913 to F. M. Ruschhaupt; 1188472 of Jun. 27, 1916 to J. B. Morton; 1197307 of Sep. 5, 1916 to F. M. Ruschhaupt; 1206076 of Nov. 28, 1916 to Oscar Altpeter; 1211382 of Jan. 9, 1917 to Oscar Altpeter; 1369911 of Mar. 1, 1921 to T. H. Brown and Haughton Brown; 1387219 of Aug. 9, 1921 to H. F. Weiss; 1467594 of Sep. 11, 1923 to H. F. Weiss; 1548763 of Aug. 4, 1925 to G. L. Sleight; 1575378 of Mar. 2, 1926 to F. R. Muller; 1593071 of Jul. 20, 1926 to R. J. Gruenberg; 1732281 of Oct. 22, 1929 to Thomas Robinson; 1751949 of Mar. 25, 1930 to Thomas Robinson; 2075058 of Mar 30, 1937 to Thomas Robinson; **Brit. Pats.** of 1872 (Apr. 27), 1264 to W. A. Lyttle; 353919 of Apr. 25, 1930 to C. M. C. Hughes and Rubberphalte, Ltd.; 445811 of Mar. 29, 1935 to United Limmer & Verwohle Rock Asphalte Co., Ltd.; **Ger. Pat.** 509042 of Jun. 13, 1926 to Alexander Wendler.

189 [p. 554] **U. S. Pat.** 1581308 of Apr. 20, 1926 to S. B. Christy, O. M. Long and F. R. Brents.

190 [p. 555] **U. S. Pats.** 1263847 of Apr. 23, 1918 to T. H. Brown; 1549867 of Aug. 18,

1925 to W. H. Gravemen; 1650709 of Nov. 29, 1927 to W. H. Gravemen; 1698242 of Jan. 8, 1929 to Thomas Robinson; **Brit. Pat.** of 1877 (Jun. 8), 2236 to A. M. Clark.

191 [p. 555] **U. S. Pat.** 1743744 of Jan. 14, 1930 to H. F. Winkelmann.

192 [p. 555] **Ger. Pat.** 118961 of Jun. 20, 1900 to Paul Sohège.

193 [p. 555] **U. S. Pat.** 1949229 of Feb. 27, 1934 to F. W. Yeager.

194 [p. 555] **U. S. Pats.** 1197307 of Sep. 5, 1916 to F. M. Ruschhaupt; 1575378 of Mar. 2, 1926 to F. R. Muller; 1643059 of Sep. 20, 1927 to F. C. J. de Both; 1767532 of Jun. 24, 1930 to Lester Kirschbraun; 1894394 of Jan. 17, 1933 to Davis Brown; **Ger. Pat.** 569544 of Nov. 1, 1931 to Bosari & Co.; **French Pat.** 542505 of Oct. 19, 1921 to F. C. J. de Both.

195 [p. 555] **U. S. Pat.** 1821552 of Sep. 1, 1931 to Lester Kirschbraun; **Can. Pat.** 311314 of May 12, 1931 to Patent & Licensing Corp.; **Ger. Pat.** Appl. D–57249 of Dec. 12, 1928 to J. H. Ditter.

196 [p. 555] **Belgium Pat.** 375500 of Jan. 31, 1931 to R. de Cuyper.

197 [p. 555] **Ger. Pat.** 75499 of May 27, 1893 to H. I. Noke.

198 [p. 555] "Paper Tiles," by Anon., *Scientific American*, **53** (New Series), No. 18, 272 (1885); **U. S. Pat.** 1751949 of Mar. 25, 1930 to Thomas Robinson; **Can. Pats.** 322097 of May 3, 1932 to Lancaster Asphalt, Inc.; 324790 of Aug. 2, 1932 to Lancaster Asphalt, Inc.; 328679 of Dec. 20, 1932 to Lancaster Asphalt, Inc.

199 [p. 555] **U. S. Pat.** 1521128 of Dec. 30, 1924 to Thomas Robinson.

200 [p. 555] **U. S. Pat.** 1799500 of Apr. 7, 1931 to T. D. Brophy.

201 [p. 555] **U. S. Pat.** 1858224 of May 10, 1932 to A. C. Fischer; **Can. Pat.** 355933 of Feb. 11, 1936 to Ruberoid Co., Ltd.; **Brit. Pat.** 444607 of Mar. 24, 1936 to Ruberoid Co., Ltd.

202 [p. 555] **Ger. Pat.** Design 1324285 of Dec. 14, 1934 to Hermann Baethge.

203 [p. 555] "Proposed Commercial Standard for Asphalt Tile," TS-2007, issued Apr. 10, 1934 (based on Proposed Federal Spec. dated Dec. 7, 1933).

204 [p. 555] **U. S. Pat.** 1430392 of Sep. 26, 1922 to A. R. Lukens; 1985201 of Dec. 18, 1934 to J. F. Maguire; 2022707 of Dec. 3, 1935 to Edmund Claxton and M. K. Bare.

205 [p. 555] **U. S. Pats.** 1637301 and 1637302 of Jul. 26, 1927 to G. C. Hannam and J. W. Schede.

206 [p. 555] **Swiss Pat.** 75777 of Jun. 19, 1917 to C. F. Weber, A.-G.

207 [p. 555] **U. S. Pat.** 1961613 of Jun. 5, 1934 to Johann Lessnig.

208 [p. 555] **U. S. Pat.** 1319918 of Oct. 28, 1919 to C. L. V. Zimmer; **Brit. Pat.** 366304 of Feb. 20, 1931 to Nicolaas van der Willik.

209 [p. 555] **U. S. Pats.** 1942060 of Jan. 2, 1934 to G. P. Heppes; 2010017 of Aug. 6, 1935 to G. P. Heppes; Reissue 19747 of Nov. 5, 1935 to G. P. Heppes.

210 [p. 555] **U. S. Pat.** 1796921 of Mar. 17, 1931 to F. C. J. de Both.

211 [p. 555] "Federal Spec. for Tile; Asphalt," Federal Standard Stock Catalogue, Section IV (Part 5), SS-T-306, May 1, 1934; "Indentation of Asphalt Tile," by J. W. McBurney, *Proc. A.S.T.M.*, 34, Part II, 591 (1934).

212 [p. 556] **Ger. Pat.** 119084 of Sep. 29, 1899 to Hugo Beck.

213 [p. 556] **U. S. Pats.** 1552852 of Sep. 8, 1925 to H. C. Innes; 1828193 of Oct. 20, 1931 to H. L. Levin; **Can. Pat.** 304583 of Oct. 7, 1930 to H. L. Levin.

214 [p. 556] **U. S. Pat.** 1304483 of May 20, 1919 to Heisaburo Inoue.

215 [p. 556] **U. S. Pats.** 1778727 of Oct. 21, 1930 to A. I. Rankin; 1925460 of Sep. 5, 1933 to J. C. Pegram.

216 [p. 556] **U. S. Pats.** 1852696 of Apr. 5, 1932 to S. W. Chaffee; Reissue 19518 of Apr. 9, 1935 to S. W. Chaffee.

217 [p. 556] **U. S. Pat.** 2020576 of Nov. 12, 1935 to K. R. Runde.

218 [p. 557] **U. S. Pat.** 1907417 of May 2, 1933 to A. C. Fischer.

219 [p. 557] **Can. Pat.** 363713 of Jan. 26, 1937 to Johns-Manville Corp.; **Brit. Pat.** 138865 of Feb. 14, 1919 to Norton Co.; **Ger. Pat.** 126435 of Nov. 17, 1899 to J. C. Anderson.

220 [p. 557] **U. S. Pats.** 1266846 of May 21, 1918 to Juan Mugica; 1688138 of Oct. 16, 1928 to W. H. Droege; 1699705 of Jan. 22, 1929 to Marcel Levy; **Can. Pat.** 302424 of Jul. 29, 1930 to W. H. Droege.

221 [p. 557] **U. S. Pats.** 300464 of Jun. 17, 1884 to Levi Haas; 855792 of Jun. 4, 1907 to J. E. Lappen; 915860 of Mar. 23, 1909 to H. L. Hollister; 934030 of Sep. 5, 1915 to F. A. Lobert; 1353619 of Sep. 21, 1920 to H. W. White; 1454823 of May 8, 1923 to H. A. Smith; 1461337 of Jul. 10, 1923 to H. F. Weiss; Reissue 15949 of Nov. 18, 1924 to H. W. White; 1760713 of May 27, 1930 to G. F. A. Ochs; 1873887 of Aug. 23, 1932 to O. A. Heppes; 1882702 of Oct. 18, 1932 to J. D. Alley; **Brit. Pat.** of 1875 (Jun. 9), 4267 to Alexander Wilkinson.

222 [p. 557] **U. S. Pat.** 1383749 of Jul. 5, 1921 to R. G. Osborne.

223 [p. 557] **U. S. Pat.** 1729172 of Sep. 24, 1929 to A. P. Lundin; **Can. Pat.** 317906 of Dec. 8, 1931 to Warren Brothers Co.

224 [p. 557] **Ger. Pats.** Appl. M–108369 of Jan. 1, 1929 to B. E. Monteux and P. L. Durand; Appl. M–192.30 of Jul. 17, 1930 to B. E. Monteux and P. L. Durand.

225 [p. 557] **U. S. Pats.** 210204 of Nov. 26, 1878 to Adolphe Kempenner; 282914 of Aug. 7, 1883 to J. F. Martin; 361347 of Apr. 19, 1887 to C. T. Crowell; 361759 of Apr. 26, 1887 to S. H. Gilson: 702140 of Jun. 10, 1902 to G. L. R. de Lenchères; 1452309 and 1452310 of Apr. 17, 1923 to Gustav Monrath; 1476828 of Dec. 11, 1923 to Gustav Monrath.

226 [p. 557] **U. S. Pats.** 506356 of Oct. 10, 1893 to G. W. Bedbury; 516999 of Mar. 20, 1894 to W. L. Carter and O. C. Hinman; **Brit. Pat.** of 1910 (May 18), 12224 to J. Heilpern.

227 [p. 557] **U. S. Pat.** 284794 of Sep. 11, 1883 to S. M. Allen; **Ger. Pat.** 316395 of Sep. 19, 1917 to Gustav Busch.

228 [p. 557] **U. S. Pat.** 1776468 of Sep. 23, 1930 to M. C. Frank; **Ger. Pat.** 541439 of Jun. 20, 1931 to Michael Groskopf.

229 [p. 557] "Handbuch der ang. Chemie Deutschland," by Engelhart and Alex, p. 502 (1846); **Ger. Pat.** 36666 of Dec. 15, 1885 to C. F. W. Doehring.

230 [p. 557] **U. S. Pats.** 41284 of Jan. 19, 1864 to D. W. Denton; 1478769 of Dec. 25, 1923 to W. E. Sheene; **Can. Pat.** 328981 of Jan. 3, 1933 to W. W. Haase.

231 [p. 557] **U. S. Pat.** 1887368 of Nov. 8, 1932 to E. E. Zech.

232 [p. 557] **U. S. Pat.** 2038120 of Apr. 21, 1936 to F. J. Mead; **Can. Pat.** 360446 of Sep. 8, 1936 to Alex Begun and R. W. Wise; 362707 of Dec. 15, 1936 to J. W. Prince.

233 [p. 557] "The Gramophone Record," by H. C. Bryson, Ernest Benn, Ltd., London (1935); **U. S. Pats.** 1134774 of Apr. 6, 1915 to F. W. Thomas; 1516469 of Nov. 18, 1924 to J. S. Miller, Jr.; **Brit. Pat.** 351175 of May 14, 1929 to A. V. y de Rueda; **Ger. Pat.** 227208 of Jun. 20, 1909 to Wilhelm Winter and Ernst Winkler.

234 [p. 557] **Brit. Pat.** 290801 of Apr. 19, 1927 to E. Noack; **French Pat.** 633790 of May 3, 1927 to Polyphonweke, A.-G.

235 [p. 557] **U. S. Pat.** 303301 of Aug. 12, 1884 to Carl Lortzing; 1409301 of Mar. 14, 1922 to William King and W. L. King; **Ger. Pats.** 293871 of Sep. 1, 1914 to Wilhelm Reiner; 294050 of Feb. 13, 1916 to Wilhelm Reiner.

236 [p. 558] "Briquetting," by A. L. Stillman, The Chemical Publishing Co., Easton, Pa. (1923); **U. S. Pat.** 1123719 of Jan. 5, 1915 to Carl Fohr.

237 [p. 558] "Briquetting and Tar Distilling Plant of the Nürnberg Gas Works," by Rudolf Terhaerst, *J. Gasbel.*, 58, 300 (1915).

238 [p. 558] "Binders for Coal Briquettes," by J. E. Mills, Bull. 24, Bureau of Mines, Wash., D. C. (1911); "Fuel-Briquetting Investigations, July 1904 to July 1912," by C. L. Wright, Bull. 58, Bureau of Mines, Wash., D. C. (1913). **U. S. Pat.** 1890349 of Dec. 6, 1932 to J. M. Weiss.

239 [p. 558] **U. S. Pat.** 1603961 of Oct. 19, 1926 to Lucien Liais.

240 [p. 558] **Brit. Pat.** of 1913 (Oct. 8), 22738 to D. J. Davis, J. T. Armstrong and James Mordan.

241 [p. 558] **Can. Pat.** 340912 of Apr. 17, 1934 to Briquet Engineering Co.

242 [p. 558] **Ger. Pat.** 286549 of Nov. 8, 1913 to Barrett Mfg. Co.

243 [p. 558] "Navy Fuel Oil Specifications and Colloidal Fuel," by S. E. Sheppard, also "Colloidal Fuel," by L. W. Bates, both published by The Submarine Defense Association, New York (1921); "Colloidal Fuels, Their Preparation and Properties," by S. E. Sheppard, *J. Ind. Eng. Chem.*, 13, 37 (1921); "Colloidal Fuel," by J. L. Strevens, *Colliery Eng.*, 13, 124,

200 and 275 (1936); **U. S. Pats.** 1274480 of Aug. 6, 1918 to O. F. Werner; 1375811 of Apr. 26, 1921 to L. W. Bates; 1382456 and 1382457 of Jun. 21, 1921 to L. W. Bates; 1447008 of Feb. 27, 1923 to L. W. Bates; **Can. Pat.** 343757 of Aug. 7, 1934 to Cunard Steamship Co., Ltd.

244 [p. 559] "Bibliography of Recent Literature of Flotation of Ores," Bull. 135, Bureau of Mines, Wash., D. C. (1917); "Answers to Questions on the Flotation of Ores," by O. C. Ralston, Tech. Paper 149, Bureau of Mines, Wash., D. C. (1917); "Colloids and Flotation," by F. G. Moses, Tech. Paper 200, Bureau of Mines, Wash., D. C. (1918); "Flotation," by E. W. Mayer and Hubert Schranz, Verlag S. Hirzel, Leipzig (1931); "Flotation," by A. M. Gaudin, McGraw-Hill Book Co., Inc., New York (1932).

245 [p. 560] "Canadian Wood Oils for Ore Flotation," by R. E. Gilmore and C. S. Parsons, *J. Soc. Chem. Ind.*; **37**, 97A (1918).

246 [p. 560] **U. S. Pat.** 1457708 of Jun. 5, 1923 to Royer Luckenbach.

247 [p. 561] "Tests of Damp-proofing and Waterproofing Compounds and Materials," by R. J. Wig and P. H. Bates, Tech. Paper 3, Bureau of Standards, Wash., D. C. (Aug. 22, 1911); "Electrolytic Corrosion of Iron in Soils," by Burton McCullum and K. H. Logan, Tech. Paper 25, Bureau of Standards, Wash., D. C. (Jun. 12, 1913); "Modern Methods of Waterproofing," by M. H. Lewis, Norman W. Henley Publishing Co., New York, (1914); "Durability of Stucco and Plaster Construction," by R. J. Wig and J. C. Pearson, Tech. Paper 70, Bureau of Standards, Wash., D. C. (Jan. 31, 1917); "Tests of Integral and Surface Waterproofings for Concrete," by C. H. Jumper, *Bureau of Standards J. Research*, **7**, 1147 (1931).

248 [p. 561] "Tar and Cement Pavement," by R. Grimshaw, *Mun. Eng.*, **43**, 11 (1912); **U. S. Pats.** 1765 of Sep. 3, 1840 to Jacob Bump; 57874 of Sep. 11, 1866 to C. K. Daniel and S. F. Daniel; 777794 of Dec. 20, 1904 to H. J. Livingston; 1760214 of May 27, 1930 to Richard Schubert; **Can. Pat.** 323383 of Jun. 21, 1932 to G. S. Eldrigde; **Brit. Pat.** of 1915 (Oct. 27), 15195 to M. I. Poznanski; **Ger. Pat.** 502017 of Feb. 23, 1928 to Richard Schubert.

249 [p. 561] **U. S. Pats.** 200122 of Feb. 12, 1878 to M. B. Bailey; 862318 of Aug. 6, 1907 to J. A. Dubbs; 958063 of May 17, 1910 to J. H. Amies; 1215273 of Feb. 6, 1917 to W. E. Hassam; 1711727 of May 7, 1929 to C. N. Forrest; **Brit. Pat.** of 1910 (Dec. 28), 30091 to J. Krumpelman.

250 [p. 561] "Bitumenized Cement and Mortar," by J. Seitz, *Asphalt Teer Strassenbautech.*, **35**, 648 (1935); **U. S. Pat.** 1000545 of Aug. 15, 1911 to L. W. Page; 1283546 of Nov. 5, 1918 to H. M. Olson; **Brit. Pat.** 253963 of Feb. 21, 1925 to C. M. C. Hughes; **Austrian Pat.** 33262 of Jun. 10, 1908 to Gottfried Schruf; **Swiss Pat.** 44284 of Dec. 28, 1907 to Gottfried Schruf.

251 [p. 561] **U. S. Pats.** 1077689 of Nov. 4, 1913 to Carleton Ellis; 1912626 of Jun. 6, 1934 to Franz Drexler and Emmerich von Pongratz; **Ger. Pat.** 579386 of Jun. 24, 1933 to I. G. Farbenindustrie, A.-G.

252 [p. 561] **U. S. Pats.** 1644964 and 1644965 of Oct. 11, 1927 to H. V. Welch.

253 [p. 561] **U. S. Pats.** 1331217 of Feb. 17, 1920 to A. E. Schutte; 1589512 of Jun. 22, 1926 to A. L. Clapp; 1788748 of Jan. 13, 1931 to A. E. Schutte; 1826529 of Oct. 6, 1931 to A. E. Schutte.

254 [p. 561] **U. S. Pats.** 959620 of May 31, 1910 to W. R. Seigle; 1305645 of Jun. 3, 1919 to Samuel Cabot; 1555940 of Oct. 6, 1925 to August Bültemann; 1699705 of Jan. 22, 1929 to Marcel Lévy; Reissue 18180 of Sep. 8, 1931 to Marcel Lévy; 2026988 and 2026989 of Jan. 7, 1936 to Hermann Plauson; 2044204 of Jun. 16, 1936 to B. F. Brice; **Brit. Pat.** 381694 of Oct. 13, 1932 to André Zivy; **Ger. Pats.** 65847 of Nov. 10, 1892 to Konrad Trobach and Richard Huppertsberg; 142136 of Jun. 13, 1902 to P. V. Parsy; 438480 of Nov. 7, 1922 to P. Anft; 530545 of Oct. 26, 1929 to Ernst Goger; 609740 of Feb. 22, 1935 to Alphons Wyss-Kohler.

255 [p. 561] **U. S. Pat.** 1912626 of Jun. 6, 1933 to Franz Drexler and Emmerich von Pongratz.

256 [p. 561] **Can. Pat.** 330109 of Feb. 14, 1933 to J. M. Evans.

257 [p. 561] **U. S. Pat.** 1285636 of Nov. 26, 1918 to Donald Davidson.

258 [p. 561] "Bituminized Cements," by Walter Dyckerhoff, *Zement*, **22**, 400 and 413 (1933); *Tonind.-Ztg.*, **57**, 637 and 660 (1933); **U. S. Pats.** 1989892 of Feb. 5, 1935 to Albert Sommer; 2055224 of Sep. 22, 1936 to Albert Sommer; Reissue 20119 of Sep. 22, 1936 to Albert Sommer; 2061774 of Nov. 24, 1936 to H. M. Olson; **Can. Pats.** 338053 of Dec. 19, 1933 to "Straba" Strassenbaubedarfs A.-G.; 357673 of May 5, 1936 to Karl Halbach; **Brit. Pats.** 394122 of Feb. 24, 1932 to "Straba" Strassenbaubedarfs A.-G.; 404787 of Nov. 5, 1932 to "Straba" Strassenbaubedarfs A.-G.; 430184 of Jun. 14, 1935 to "Straba" Strassenbubeadarfs A.-G.; **Ger. Pats.** Appl. St-48984 of Feb. 24, 1932 to "Straba" Strassenbaubedarfs A.-G.; 600184 of Jul. 18, 1934 to "Straba" Strassenbaubedarfs A.-G.; 604340 of Jun. 1, 1932 to "Straba" Strassenbaubedarfs A.-G.; 616012 of Jun. 14, 1932 to "Straba" Strassenbaubedarfs A.-G.; 637300 of Oct. 27, 1936 to "Straba" Strassenbaubedarfs A.-G.

259 [p. 561] **U. S. Pat.** 1975485 of Oct. 2, 1934 to Albert Sommer.

260 [p. 562] "Some Experiments with Mortars and Concrete with Asphaltic Oils," by Arthur Taylor and Thomas Sanborn, *Proc. Am. Soc. Civil Eng.*, **39**, 355 (1913).

261 [p. 562] "Oil-mixed Portland Cement Concrete," by L. W. Page, Bull. 46, Office of Public Roads, Wash., D. C. (Aug. 8, 1912); Bull. 230, Office of Public Roads, Wash., D. C. (Jul. 14, 1915); "Integral Waterproofings for Concrete," by A. H. White, *J. Ind. Eng. Chem.*, **15**, 150 (1923).

262 [p. 563] "Water-soluble Coal Tar," by Friedrich Raschig, *Chem. Rev. Fett- u. Harz-Ind.*, **17**, 169 (1910); also *Z. angew. Chem.*, **23**, 973 (1910); **U. S. Pats.** 1049916 of Jan. 7, 1913 to Friedrich Raschig; 1959586 of May 22, 1934 to Lester Kirschbraun; 2038367 of Apr. 21, 1936 to E. P. C. Lambert; **Brit. Pat.** of 1910 (May 4), 15711 to Friedrich Raschig; **Ger. Pat.** 68532 of Jul. 1, 1891 to Grünzweig & Hartmann.

263 [p. 563] **U. S. Pats.** 1296083 of Mar. 4, 1919 to Robert Illemann; 1302810 of May 6, 1919 to Lester Kirschbraun; 1744869 of Jan. 28, 1930 to Richard Cross; **Brit. Pat.** of 1908 (Jul. 16), 15100 to Julius Kathe; **Ger. Pat.** 211877 of Sep. 5, 1906 to Julius Kathe.

264 [p. 563] **U. S. Pats.** 903287 of Nov. 10, 1908 to H. V. D. Heide; 1204259 of Nov. 7, 1916 to Ignaz Egger and Carl Gartenmann; **Can. Pat.** 148987 of Jul. 1, 1913 to Ignaz Egger and Carl Gartenmann; **Brit. Pat.** of 1910 (Jul. 7), 16264 to T. D. Kelly; **Ger. Pats.** 103733 of Jan. 13, 1898 to Hans Wunner; Appl. W-27653 of Apr. 29, 1907 to Hans Wunner (rejected).

265 [p. 563] **U. S. Pats.** 1134573 of Apr. 6, 1915 to Herbert Abraham and H. W. Haines; 1242585 of Oct. 9, 1917 to H. M. Olson; 1607405 of Nov. 16, 1926 to H. S. Lukens; 1959586 of May 22, 1934 to Lester Kirschbraun; **Can. Pat.** 325817 of Sep. 6, 1932 to "Straba" Strassenbaudedarfs A.-G.; **Brit. Pats.** 388093 of Feb. 23, 1933 to Robert Tagg; 438188 of May 13, 1935 to J. R. Geigy A.-G.; **Ger. Pat.** 625490 of Feb. 10, 1936 to J. R. Geigy A.-G.; **French Pat.** 789867 of Nov. 7, 1935 to J. R. Geigy, S. A.

266 [p. 563] **U. S. Pats.** 1589512 of Jun. 22, 1926 to A. L. Clapp; 1788748 of Jan. 13, 1931 to A. E. Schutte; **Can. Pat.** 248326 of Mar. 31, 1925 to Flintkote Co.

267 [p. 563] **U. S. Pat.** 1599903 of Sep. 14, 1926 to E. C. E. Lord.

268 [p. 563] **U. S. Pat.** 1408224 of Feb. 28, 1922 to C. S. Reeve.

269 [p. 563] **U. S. Pat.** 1382986 of Jun. 28, 1921 to L. M. Kregelius.

270 [p. 563] **U. S. Pat.** 1305645 of Jun. 3, 1919 to Samuel Cabot; **Brit. Pats.** 124669 of Sep. 10, 1918 to British Westinghouse Electric & Mfg. Co.; 368800 of Nov. 5, 1930 to Hermann Plauson and Maurice Imer; 378107 of May 11, 1931 to Hermann Plauson and Maurice Imer.

271 [p. 563] **U. S. Pats.** 1109120 of Sep. 1, 1914 to Carleton Ellis; 1752214 of Mar. 25, 1930 to L. S. van Westrum; 2067772 of Jan. 12, 1937 to H. L. Levin.

272 [p. 563] **Ger. Pats.** 242454 of Nov. 16, 1910 to Paul Mecke; 248297 of Jun. 22, 1911 to Paul Mecke.

273 [p. 563] **Ger. Pat.** Appl. H-134384 of Dec. 12, 1932 to Bindemittel, G.m.b.H.

274 [p. 563] **Brit. Pat.** 322792 of Sep. 13, 1928 to Colas Products, Ltd., L. G. Gabriel and J. F. Blott.

275 [p. 563] **U. S. Pat.** 1889437 of Nov. 29, 1932 to A. J. de Lange; **Can. Pat.** 318861 of Jan. 12, 1932 to Flintkote Co.

276 [p. 563] **Can. Pat.** 313361 of Jul. 21, 1931 to C. D. Cory.

277 [p. 563] **U. S. Pat.** 1772999 of Aug. 12, 1930 to F. W. Huber; **Can. Pats.** 318980 and 318981 of Jan. 19, 1932 to F. W. Huber.

278 [p. 563] **U. S. Pat.** 1637475 of Aug. 2, 1927 to Lewis Davis and E. W. Bennett.

279 [p. 563] **Ger. Pat.** 555394 of Jun. 28, 1930 to Friedrich Winzer.

280 [p. 563] **U. S. Pat.** 2047426 of Jul. 14, 1936 to F. E. Miller.

281 [p. 563] "Federal Spec. for Waterproofing-Material; Integral (for Use with Portland-Cement-Mortar or Concrete)," Federal Standard Stock Catalogue, Section IV (Part 5), SS-W-101, Jan. 6, 1931; "Manufacture of Bituminous Concrete and a Study of Some of its Properties," by V. Ponomarev and L. Danilushkina, Colloid J. (U. S. S. R.), 2, 171 (1936).

282 [p. 564] **U. S. Pat.** 1402412 of Jan. 3, 1922 to Karl Friedrich; **Brit. Pat.** 154236 of Mar. 20, 1918 to Karl Friedrich.

283 [p. 565] **U. S. Pat.** 1920065 of Jul. 25, 1933 to Putnam Davis.

284 [p. 565] **U. S. Pats.** 1331554 of Feb. 24, 1920 to M. K. Armstrong; 1641611 of Sep. 6, 1927 to M. K. Armstrong; Reissue 18363 of Feb. 23, 1932 to M. K. Armstrong; 1901055 of Mar. 14, 1933 to H. K. Linzell; 2031171 of Feb. 18, 1936 to H. K. Linzell; **Can. Pats.** 333216 of Jun. 13, 1933 to Canadian Gypsum Co., Ltd.; 341478 of May 8, 1934 to Canadian Gypsum Co., Ltd.; **Russian Pat.** 34372 of Jan. 31, 1934 to M. I. Khigerovich and N. N. Denilova.

285 [p. 565] **U. S. Pats.** 1607405 of. Nov. 16, 1926 to H. S. Lukens; 1659867 of Feb. 21, 1928 to Leopold Frieser; 1724945 of Aug. 20, 1929 to H. S. Lukens; 1752194 of Mar. 25, 1930 to J. U. A. Ohlsen; 1906276 of May 2, 1933 to H. S. Lukens; 1968784 of Jul. 31, 1934 to E. J. Milburn; Reissue 19779 of Dec. 3, 1935 to H. S. Lukens; 2031171 of Feb. 18, 1936 to H. K. Linzell; **Can. Pat.** 300739 of Jun. 3, 1930 to L. C. Schilling; **Brit. Pats.** of 1907 (Jul. 9), 15761 to W. G. F. Siegmann; 3038889 of Jan. 2, 1929 to L. C. Schilling; **Ger. Pats.** 211849 of Jul. 17, 1907 to Sagax Wood Co.; 593121 of Feb. 23, 1924 to L. C. Schilling.

286 [p. 565] **U. S. Pat.** 126662 of May 14, 1872 to W. W. Wilcox.

287 [p. 565] **U. S. Pats.** 1347869 of Jul. 27, 1920 to R. G. Osborne; 1401974 of Jan. 3, 1922 to A. C. Fischer; 1441904 of Jan. 9, 1923 to C. C. Bancroft.

288 [p. 565] **U. S. Pats.** 1513382 of Oct. 28, 1924 to A. C. Fischer; 1591670 of Jul. 6, 1926 to A. C. Fischer; 1603961 of Oct. 19, 1926 to Lucien Liais; 1744378 of Jan. 21, 1930 to A. C. Fischer; **Brit. Pat.** 231140 of Feb. 9, 1925 to Lucien Liais; **French Pat.** 578815 of Mar. 18, 1924 to Lucien Liais.

289 [p. 565] **U. S. Pats.** 1630103 of May 24, 1927 to J. H. Young; 1915940 of Jun. 27, 1933 to R. H. McKee; **Can. Pat.** 276488 of Dec. 20, 1927 to H. H. Robertson Co.

290 [p. 565] **U. S. Pat.** 1738906 of Dec. 10, 1929 to Lester Kirschbraun.

291 [p. 565] **Ger. Pat.** 536712 of Nov. 19, 1926 to E. B. Hack.

292 [p. 565] **U. S. Pat.** 1347869 of Jul. 27, 1920 to R. G. Osborne.

293 [p. 565] **U. S. Pats.** 1239074 of Sep. 4, 1917 to A. G. Bagnall and J. A. Taylor; 1402177 of Jan. 3, 1922 to R. P. Perry; 1441904 of Jan. 9, 1923 to C. C. Bancroft; 1698877 and 1698878 of Jan. 15, 1929 to M. R. Cone; 1818346 of Aug. 11, 1931 to O. F. Mottweiler and D. C. Drill; 2048651 of Jul. 21, 1936 to C. L. Norton; **Ger. Pat.** Appl. St–49469 of Jun. 13, 1932 to "Straba" Strassenbaubedarfs A.-G.

294 [p. 565] **U. S. Pats.** 441951 of Dec. 2, 1890 to G. W. Goetz; 1432742 of Oct. 24, 1922 to F. J. Commin; 1699536 of Jan. 22, 1929 to L. C. Jones, C. C. Loomis and H. W. Banks.

295 [p. 565] **U. S. Pats.** 1088635 of Feb. 24, 1914 to C. N. Stevens; 1210097 of Dec. 26, 1916 to R. P. Perry and E. F. Stewart; 1289892 of Dec. 21, 1918 to R. P. Perry; 1293535 of Feb. 4, 1919 to R. P. Perry; 1294909 of Feb. 18, 1919 to K. P. Howell; 1352623 of Sep. 14, 1920 to R. P. Perry.

296 [p. 565] **U. S. Pat.** 1670844 of May 22, 1928 to R. R. Cone; 2013808 of Sep. 10, 1935 to Thomas Robinson.

297 [p. 565] **U. S. Pat.** 1951790 of Mar. 20, 1934 to R. A. Curran.

298 [p. 565] **U. S. Pats.** 2006585 and 2006586 of Jul. 2, 1935 to J. S. Downard; 2063663 of Dec. 8, 1936 to J. S. Downard.

299 [p. 565] **U. S. Pat.** 2062374 of Dec. 1, 1936 to H. M. Noel; **French Pat.** 787756 of

Sep. 28, 1935 to Standard Oil Development Co.; **Ger. Pat.** Appl. St–53825 of Aug. 17, 1935 to Standard Oil Development Co.

300 [p. 565] **U. S. Pat.** 1951769 of Mar. 20, 1934 to H. P. Pearson and L. J. Burrows.

301 [p. 565] **U. S. Pat.** 1104140 of Jul. 21, 1914 to J. C. Ross; **Can. Pat.** 350654 of Jun. 4, 1935 to F. W. Atack.

302 [p. 565] **U. S. Pat.** 1781105 of Nov. 11, 1930 to J. S. Downard.

303 [p. 565] **U. S. Pat.** 1736915 of Nov. 26, 1929 to Robert Illemann and R. A. Whitson.

304 [p. 565] **U. S. Pats.** 309787 of Dec. 23, 1884 to E. Judson; 359289 of Mar. 15, 1887 to E. Schultze; 1211721 of Jan. 9, 1917 to Ludwig Landsberg.

305 [p. 565] **U. S. Pat.** 205750 of Jul. 9, 1878 to H. W. Johns.

306 [p. 566] **Ger. Pat.** 589960 of Dec. 19, 1933 to "Emulgia" Vertriebsgesellschaft für Asphalt-, Teer- und Strassenbauprodukte, Clause & Co., Kommanditgesellschaft.

307 [p. 566] "Gelatinized Tars and Bitumens," by E. Da Fano, Asphalti, bitumi, catrami, 6, 40 (1934).

CHAPTER XXVIII

1 [p. 567] DIN–1996 "Vorschriften für die Lieferung von Asphalt und Teer, sowie von Asphalt und Teer enthaltenden Massen" (B 1/2, B 1/6, B 1/33) 1929; DIN–1991 "Grundsätze für die Herstellung und Unterhaltung von Asphaltstrassen, allgemeine Vorschriften" (B 1/6, B 1/33), 1928; DIN–1992 "Sondervorschriften für Stampfasphalt- und Hartgussasphaltstrassen" (B 1/6), 1928; DIN–1993 "Sondervorschriften für Walzasphaltstrassen," 1928; * * * * All the foregoing published by Beuth-Verlag, Berlin ("Deutscher Normenausschuss.") "Typical Specifications for Bituminous Road Materials," by Prevost Hubbard and C. S. Reeve, Bull. No. 691, U. S. Dept. of Agriculture, Wash., D. C. (Jul. 10, 1918); "Ueber neuere Kaltasphaltverfahren im Strassenbau," by Walter Obst, *Teer u. Bitumen*, 27, 377 (1929); "Standard Definitions of Terms Relating to Materials for Roads and Pavements" (D 8–33), A.S.T.M. Standards 1936, II, 1116.

2 [p. 570] **Ger. Pat.** 614037 of May 31, 1935 to J. R. Geigy A.-G.

3 [p. 570] **U. S. Pat.** 938698 of Nov. 2, 1909 to J. A. W. Pine; 1188081 of Jun. 20, 1916 to Lester Kirschbraun.

4 [p. 568] "Federal Spec. for Asphalt; Cut-Back (for) Road-Work," Federal Standard Stock Catalogue, Section IV (Part 5), SS-A-671, Dec. 5, 1933.

5 [p. 568] "Surface Treatment Types," Manual No. 2, Asphalt Road Costruction Series, The Asphalt Institute, New York (1932).

6 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 10 to 15 Penetration, for the Manufacture of Asphalt Block" (D 133–23T), A.S.T.M. Tentative Standards 1936, 672.

7 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 15 to 25 Penetration, for the Manufacture of Asphalt Block," (D 134–23T), A.S.T.M. Tentative Standards 1936, 674.

8 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 25 to 30 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements," (D 163–23T), A.S.T.M. Tentative Standards 1936, 676.

9 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 30 to 40 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements," (D 164–23T), A.S.T.M. Tentative Standards 1936, 678.

10 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 40 to 50 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements and as Filler for Brick and Block Pavements," (D 99–26T), A.S.T.M. Tentative Standards 1936, 680.

11 [p. 570, insert] "Federal Spec. for Asphalt; (for Use in) Road and Pavement Construction," Federal Standard Stock Catalogue, Section IV (Part 5), SS-A-706 (Jun. 23, 1931).

12 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 50 to 60 Penetration, for Use in Sheet Asphalt and Asphaltic-Concrete Pavements and as Filler for Block Pavements," (D 100–26T), A.S.T.M. Tentative Standards 1936, 682.

13 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 60 to 70 Penetration, for Use in Sheet Asphalt, Asphaltic-Concrete, and Asphalt-Macadam Pavements and as Filler for Block Pavements," (D 101–26T), A.S.T.M. Tentative Standards 1936, 684.

14 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 85 to 100 Penetration, for Use in Asphalt-Macadam Pavements," (D 102–24T), A.S.T.M. Tentative Standards 1936, 686.

15 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 100 to 120 Penetration, for Use in Asphalt-Macadam Pavements," (D 103–24T), A.S.T.M. Tentative Standards 1936, 688.

16 [p. 570, insert] "Tentative Specifications for Asphalt Cement, 120 to 150 Penetration, for Use in Asphalt-Macadam Pavements," (D 135–23T), A.S.T.M. Tentative Standards 1936, 690.

17 [p. 570, insert] "Tentative Specifications for Asphalt Filler for Brick Pavements (Blown Type)," (D 241–26T), A.S.T.M. Tentative Standards 1936, 692.

18 [p. 570, insert] "Federal Spec. for Asphalt; Petroleum, Type PAF 1–25 (for) Joint-Filler (Squeegee or Pouring Method)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS–A–696 (Jun. 23, 1931).

19 [p. 570, insert] "Federal Spec. for Tar; Cold-application (for Roads)," Federal Standard Stock Catalogue, Section IV, (Part 5), R–T–101, (Jun. 23, 1931).

20 [p. 570, insert] "Federal Spec. Tar; Refined, Mixed-in-Place Construction (for) Roads," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-136 (Mar. 5, 1935).

21 [p. 570, insert] "Tentative Specifications for High-Carbon Tar for Surface Treatment, Cold Application," (D 104–36T), A.S.T.M. Tentative Standards 1936, 730.

22 [p. 570, insert] "Tentative Specifications for Low-Carbon Tar for Surface Treatment, Cold Application," (D 105–36T), A.S.T.M. Tentative Standards 1936, 732.

23 [p. 570, insert] "Federal Spec. for Tar; Refined, Hot-Application (for Roads)," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-131 (Jun. 23, 1931).

24 [p. 570, insert] "Tentative Specifications for High-Carbon Tar for Surface Treatment, Hot Application," (D 108–36T), A.S.T.M. Tentative Standards 1936, 734.

25 [p. 570, insert] "Tentative Specifications for Low-Carbon Tar for Surface Treatment, Hot Application," (D 109–36T), A.S.T.M. Tentative Standards 1936, 736.

26 [p. 570, insert] "Federal Spec. for Tar; Repair-Work (for Roads)," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-141, (Jun. 23, 1931).

27 [p. 570, insert] "Tentative Specifications for High-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product)," (D 106-28T), A.S.T.M. Tentative Standards 1936, 726.

28 [p. 570, insert] "Tentative Specifications for Low-Carbon Tar Cement for Use Cold in Repair Work (Cut-Back Product)," (D 107–28T), A.S.T.M. Tentative Standards 1936, 728.

29 [p. 570, insert] "Federal Spec. for Tar; Refined, Construction (for Roads)," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-121, (Jun. 23, 1931).

30 [p. 570, insert] "Tentative Specifications for High-Carbon Tar Cement," (D 110–36T), A.S.T.M. Tentative Standards 1936, 722.

31 [p. 570, insert] "Tentative Specifications for Low-Carbon Tar Cement," (D 111–36T), A.S.T.M. Tentative Standards 1936, 724.

32 [p. 570, insert] "Standard Specifications for Coal-Tar Pitch for Stone Block Filler," (D 112–30), A.S.T.M. Standards 1936, II, 1019.

33 [p. 570, insert] "Federal Spec. for Tar; Refined, (for) Crack-Filler," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-126, (Feb. 7, 1933).

34 [p. 570, insert] "Federal Spec. for Tar; (for) Joint-Filler," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-111 (Feb. 7, 1933).

35 [p. 570] **U. S. Pat.** 1366572 of Jan. 25, 1921 to L. M. Law.

36 [p. 571] "Die Verarbeitung von paraffinhaeltigen Rohölen zu guten Strassenasphalten," by Joachim Hausman, *Petroleum*, **28**, 1 (1932).

37 [p. 571] "Cut-back Asphalts; Their Characteristics and Use," by Prèvost Hubbard, Tenth Annual Paving Conference, New Orleans, La., (Dec. 5–9, 1932); "Single-Coat Asphalt

(Cold Process)," British Standard Spec. No. 510–1933, British Standards Institution; "Two-Coat Asphalt (Cold Process)," British Standard Spec. No. 511–1933, British Standards Institution.

38 [p. 572] "Bitumenemulsionen in Strassenbau," by Albrecht von Skopnik, *Teer u. Bitumen*, **25**, [4] (1927); "Ueber die Anwendung von Emulsionen im Strassenbau," by F. Leiter, *Teer u. Bitumen*, **26**, [7] (1928); "Ist die Bevorzugung von Teer- und Bitumenemulsionen gegenüber der Verwendung von Heissteer und Heissbitumen wirtschaftlich gerechtfertigt?", by H. Hentrich, *Teer u. Bitumen*, **27**, [1] (1929); "Teere und Bitumina im Dienste des praktischen Strassenbaues," by Albrecht von Skopnik, *Teer u. Bitumen*, **27**, [7] (1929); "Ueber Humussäure als Emulgator in Bitumenemulsionen," by Leo Eck, *Teer u. Bitumen*, **27**, [7](1929); "Teer und Bitumen in kolloidchemischer Betrachtungsweise," by H. Grohn, *Teer u. Bitumen*, **27**, [20] (1929); "Ueber neue Kaltasphaltverfahren im Strsssenbau," by W. Ost, *Teer u. Bitumen*, **27** [22] (1929); "Erfahrungen der letzten Jahre im bituminösen Strassenbau," by Albrecht von Skopnik, *Teer u. Bitumen*, **28**, [20] (1930); "Die Entwicklung der deutschen technischen Emulsionpatente für den bituminösen Strassenbau," by Albrecht von Skopnik, *Teer u. Bitumen*, **28**, 581 (1930); British Standard Spec. No. 434, "Asphaltic Bitumen Road Emulsion," British Standards Institution, London (1931); British Standard Spec. No. 618, "Emulsions of Road Tar and Road Tar-Asphaltic Bitumens," British Standards Institution, London (1935); "Merkblatt über die Verwendung von Bitumenemulsionen bei der Herstellung von Strassenbefestigungen," by Forschungsgesellschaft für das Strassenwesen, e.V., Berlin (1935); **Ger. Pat.** 173639 of Oct. 2, 1903 to L. S. van Westrum.

39 [p. 572] "Road Emulsions," by P. E. Spielmann, *Munic. Eng. Sanit. Record*, **84**, 624 (1929); **U. S. Pat.** 1884795 of Oct. 25, 1932 to C. L. McKesson.

40 [p. 573] "Tentative Specifications for Emulsified Asphalt (for Coarse Aggregate Plant Mixes)" (D 397–36T), A.S.T.M. Tentative Standards 1936, 704; American Association State Highway Officials Standard A.A.S.H.O. No: M–47; "Tentative Specifications for Emulsified Asphalt (for Retread and Coarse Aggregate Mixes)" (D 398–36T), A.S.T.M. Tentative Standards 1936, 706; American Association State Highway Officials Standard A.A.S.H.O. No: M–48; "Tentative Specifications for Emulsified Asphalt (Heavy Premix-Summer Grade)" (D 399–36T), A.S.T.M. Tentative Standards 1936, 708; American Association State Highway Officials Standard A.A.S.H.O. No: M–49; "Tentative Specifications for Emulsified Asphalt (Heavy Premix-Winter Grade)" (D 400–34T), A.S.T.M. Tentative Standards 1935, 820; American Association State Highway Officials Standard A.A.S.H.O. No: M–50; "Tentative Specifications for Emulsified Asphalt (Quick-Setting for Penetration and Surface Treatment)" (D 401–36T), A.S.T.M. Tentative Standards 1936, 710; American Association State Highway Officials Standard A.A.S.H.O. No: M–51; "Federal Spec. for Asphalt; Emulsion (for) Road Work," SS-A-674, Federal Standard Stock Catalogue, Section IV, (Part 5), May 7, 1935.

41 [p. 573] "Cold Asphalt Macadam, Penetration (Grouting and Semi-Grouting) Method, Using Road Emulsion," British Standard Spec. No. 433–1931, British Standards Institution; "Emulsified Asphalt in Road and Plant-Mixed Pavements," by C. L. McKesson, Tenth Annual Paving Conference, New Orleans, La., (Dec. 5–9, 1932); **Ger. Pat.** 490082 of Aug. 20, 1926 to Eduard Preé and F. H. Lehnert.

42 [p. 573] **Can. Pat.** 301680 of Jul. 1, 1930 to Flintkote Co.; **Ger. Pat.** 507297 of Nov. 9, 1927 to W. H. Schmitz.

43 [p. 573] **Ger. Pats.** 521281 of Oct. 22, 1926 to Eduard Preé and F. H. Lehnert; 524088 of Aug. 25, 1928 to Georg Wieland.

44 [p. 573] **Ger. Pat.** 535350 of Sep. 29, 1928 to Deutsche Vitalit, G.m.b.H.

45 [p. 573] **Ger. Pats.** 575105 of Apr. 24, 1933 to A. F. Malchow, A.-G.; 575397 of Apr. 27, 1933 to Michael Groskopf.

46 [p. 573] **Can. Pat.** 328483 of Dec. 13, 1932 to International Bitumen Emulsions Corp.

47 [p. 573] **U. S. Pat.** 1928303 of Sep. 26, 1933 to G. H. Alvey; **Brit. Pat.** 406563 of Jul. 11, 1933 to Barber Asphalt Co.

48 [p. 574] "Standard Definition of the Term Sand," (C 58–28), A.S.T.M. Standards 1936, II, 366; "Tentative Definition of the Term Aggregate" (C 58–28 T), A.S.T.M. Tentative Standards 1936, 518.

49 [p. 574 insert and 576] "Standard Specifications for Broken Slag for Bituminous Macadam Wearing Course" (D 159–27), A.S.T.M. Standards 1936, II, 1028.

50 [p. 574 insert and 576] "Standard Specifications for Broken Stone for Bituminous Macadam" (D 192–29), A.S.T.M. Standards 1936, II, 1036.

51 [p. 574 insert and 576] "Standard Specifications for Broken Slag for Bituminous Macadam Base" (D 195–27), A.S.T.M. Standards 1936, II, 1026.

52 [p. 574 insert and 576] "Tentative Specifications for Commercial Sizes of Broken Stone and Broken Slag for Highway Construction" (D 63–23T), A.S.T.M. Tentative Standards 1936, 719.

53 [p. 574 insert and 576] "Form of Specifications for Certain Commercial Grades of Broken Stone" (D 35–18), A.S.T.M. Standards 1936, II, 1113.

54 [p. 574 insert and 576] "Asphalt Pocket Reference for Engineers" (5th Edition, Revised), The Asphalt Association, New York (1929).

55 [p. 574 insert and 576] "Federal Spec. for Crushed Stone and Crushed Slag; (for) Bituminous-Macadam Base or Surface Course," SS-C-736, Federal Standard Stock Catalogue, Section IV, (Part 5), Jan. 5, 1932.

56 [p. 574 insert and 576] "Tentative Specifications for Broken Stone for Bituminous Concrete Base" (D 193–29T), A.S.T.M. Tentative Standards 1936, 715.

57 [p. 574 insert and 576] "Standard Specifications for Broken Slag for Bituminous Concrete Base" (D 196–27), A.S.T.M. Standards 1936, II, 1022.

58 [p. 574 insert and 576] "Tentative Specifications for Commercial Sizes of Sand and Gravel for Highway Construction" (D 64–20T), A.S.T.M. Tentative Standards 1936, 713.

59 [p. 574 insert and 576] "Standard Specifications for Gravel for Bituminous Concrete Base" (D 309–30), A.S.T.M. Standards 1936, II, 1017.

60 [p. 574 insert and 576] "Federal Spec. for Crushed Stone and Crushed Slag; (for) Bituminous Concrete Base or Surface Course" SS–C–731, Federal Standard Stock Catalogue. Section IV, (Part 5), Jan. 5, 1932.

61 [p. 574 insert and 576] "Standard Specifications for Broken Slag for Bituminous Concrete (Coarse-Graded Aggregate Type)" (D 160–27), A.S.T.M. Standards 1936, II, 1023.

62 [p. 574 insert and 576] "Standard Specifications for Broken Stone for Bituminous Concrete Surface" (D 194–29), A.S.T.M. Standards 1936, II, 1034.

63 [p. 574 insert and 576] "Federal Spec. for Crushed-Stone and Crushed-Slag; (for) Binder Course, Sheet-Asphalt-Pavement" SS–C–726, Federal Standard Stock Catalogue, Section IV, (Part 5), Jan. 5, 1932.

64 [p. 574 insert and 576] "Federal Spec. for Crushed Stone, Crushed Slag, and Gravel: (for) Bituminous-Surface Treatment" SS-C-741, Federal Standard Stock Catalogue, Section IV, (Part 5), Jan. 5, 1932.

65 [p. 574 insert and 576] "Standard Specifications for Broken Slag for Bituminous Concrete (Fine-Graded Aggregate Type)" (D 161–27), A.S.T.M. Standards 1936, II, 1025.

66 [p. 574 insert and 576] "Federal Spec. for Materials; (for) Cushion Course, Brick, Stone-Block, or Wood-Block Pavements (Sand, Slag, Limestone, Screenings, etc.)" SS-M-51, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 23, 1931.

67 [p. 574 insert and 576] "Federal Spec. for Sand; (for) Cement-Mortar-Bed (for) Brick, Stone-Block, or Wood-Block Pavements" SS–S–51, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 5, 1934.

68 [p. 574 insert and 576] "Federal Spec. for Sand; (for Use in) Sheet Asphalt or Bituminous Concrete Pavements" SS–S–71, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 23, 1931.

69 [p. 574 insert and 576] "Standard Specifications for Sand for Sheet Asphalt and Bituminous Concrete Pavements" (D 162–29), A.S.T.M. Standards 1936, II, 1021.

70 [p. 574 insert and 576] "Federal Spec. for Sand; (for) Grout-Filler, Brick and Stone-Block Pavements" SS-S-61, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 23, 1931.

71 [p. 574 insert and 576] "Tentative Specifications for Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements" (D 242-26T), A.S.T.M. Tentative Standards 1936, 712.

72 [p. 574 insert and 576] "Federal Spec. for Mineral-Filler; (for) Sheet-Asphalt or Asphaltic Concrete Pavements (Portland Cement, Limestone Dust and Dolomite Dust)" SS-M-351, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 23, 1931.

73 [p. 575] "Standard Method of Test for Abrasion of Rock," (D 2-33), A.S.T.M. Standards 1936, II, 1040.

74 [p. 575] "Standard Method of Test for Toughness of Rock," (D 3-18), A.S.T.M. Standards 1936, II, 1114; approved as "American Standard" (A.S.A. No.: A 5-1930) by The American Standards Association.

75 [p. 575] "Standard Method of Test for Unit Weight of Aggregate for Concrete," (C 29-27), A.S.T.M. Standards 1936, II, 364; American Assoc. State Highway Officials' Standard A.A.S.H.O. No. T-19.

76 [p. 577] "Quality of Oil for Surface Oiling of Earth Roads and Streets," by F. L. Sperry, *Proc. A.S.T.M.*, **25**, II, 376 (1925).

77 [p. 578] "Cold Tars," by Heinrich Mallison, *Gas J.*, **202**, 862 (1933).

78 [p. 578] "Asphalt Road Construction," Manual No. 2, "Surface Treatment Types," The Asphalt Institute, New York (1932); British Standard Spec. No. 510, "Single-coat Asphalt (Cold Process)," British Standards Institution, London (1933); British Standard Spec. No. 511, "Two-coat Asphalt (Cold Process)," British Standards Institution, London (1933).

79 [p. 580] "Spec. for Asphalt Surface Treatment or Retreatment of Old Bituminous Surfaces," Spec. S-1, The Asphalt Institute, New York (Revised May 15, 1934).

80 [p. 581] "Asphalt Road Construction," Manual No. 1, "Road-Mix Types" (2nd Edition, Revised), The Asphalt Institute, New York (1932).

81 [p. 581] "Spec. for Asphaltic Road-Mix Surface Course (Macadam Aggregate Type)," Spec. R.M.-1, The Asphalt Institute, New York (Revised May 15, 1934).

82 [p. 582] "Spec. for Asphaltic Road-Mix Surface Course (Dense Graded Type)," Spec. R.M.-2, The Asphalt Institute, New York (Revised May 15, 1934).

83 [p. 583] "Asphalt Macadam (Penetration Method)," British Standard Spec. No. 347-1928 (Add. Jul. 1930), British Standards Institution; "Asphaltic Bitumen Road Emulsion for Penetration (Grouting and Semi-Grouting) and Surface Dressing," British Standard Spec. No. 434-1935, British Standards Institution; "Emulsions of Road Tar and of Road Tar-Asphaltic Bitumen Mixtures for Penetration (Grouting and Semi-Grouting) and Surface Dressing," British Standard Spec. No. 618-1935, British Standards Institution.

84 [p. 583] "Asphalt Road Construction—Road-Mix Types," Manual No. 1, published by the Asphalt Association, New York (1931).

85 [p. 583] "Asphalt Macadam Base," Spec. B-7, issued by the Asphalt Association, New York (Jan. 1, 1924); "Spec. for Asphalt Macadam Base (Black Base), (Penetration Method with Hot Asphalt Cement)," Spec. B-7, The Asphalt Institute, New York (Revised May 15, 1934).

86 [p. 584] "Asphalt Macadam Surface Course," Spec. A-1, issued by The Asphalt Association, New York, (Jan. 1, 1924); "Spec. for Asphalt Macadam Surface Course (Penetration Method with Hot Asphalt Cement)," Spec. A-1, The Asphalt Institute, New York (Revised May 15, 1934).

87 [p. 587] **U. S. Pats.** 2023068 of Dec. 3, 1935 to W. H. Flood; 2062348 of Dec. 1, 1936 to C. M. Baskin.

88 [p. 587] "Asphalt Concrete Base." Spec. B-8, issued by The Asphalt Association, New York; (Jan. 1, 1921); "Spec. for Asphaltic Concrete Base (Black Base) (Coarse Graded Aggregate Type)," Spec. B-8, The Asphalt Institute, New York (Revised May 15, 1934).

89 [p. 588] "Asphaltic Concrete Surface Course—Coarse-graded Aggregate Type,"

Spec. A-2, issued by The Asphalt Association, New York, (Jan. 1, 1921); "Spec. for Asphaltic Concrete Surface Course (Coarse Graded Aggregate Type)." Spec. A-2, The Asphalt Institute, New York (Revised May 15, 1934).

90 [p. 588] "Asphaltic Concrete Surface Course—Fine-graded Aggregate Type." Spec. A-3, issued by The Asphalt Association, New York, (Jan. 1, 1921); "Spec. for Stone Filled Sheet Asphalt Surface Course," Spec. A-3, The Asphalt Institute, New York (Revised May 15, 1934).

91 [p. 589] **U. S. Pat.** 1985572 of Dec. 25, 1934 to D. W. Johnston.

92 [p. 589] **U. S. Pat.** 1842139 of Jan. 19, 1932 to G. W. Alsdorf.

93 [p. 589] British Standard Spec. No. 348, "Compressed Rock Asphalt," British Standards Institutition, London (1935); British Standard Spec. No. 594, "Rolled Asphalt (Fluxed Lake Asphalt and Asphaltic Bitumen. Hot Process)," British Standards Institution, London (1935); British Standard Spec. No. 595, "Rolled Asphalt (Fluxed Natural Asphalt and Asphaltic Bitumen. Hot Process)," British Standards Institutition, London (1935).

94 [p. 590] "Federal Spec. for Coarse-Aggregate; (for) Portland Cement-Concrete-Pavement or Base (Crushed Stone, Gravel, or Slag)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-C-571, (Feb. 3, 1931).

95 [p. 590] **U. S. Pats.** 1302810 of May 6, 1919 to Lester Kirschbraun; 1767533 of Jun. 24, 1930 to Lester Kirschbraun; **Can. Pat.** 256079 of Dec. 8, 1925 to Lester Kirschbraun; **Brit. Pats.** 322793 of Sep. 13, 1928 to Colas Products, Ltd., L. G. Gabriel and J. F. Blott; 322835 of Oct. 3, 1928 to F. B. Dehn.

96 [p. 590] "Sheet Asphalt Binder and Surface Course," Spec. A-4, issued by The Asphalt Association, New York (Jan. 1, 1921); "Spec. for Sheet Asphalt Binder and Surface Courses," Spec. A–4, The Asphalt Institute, New York (Revised May 15, 1934); "Rolled Asphalt; Fluxed Lake Asphalt and Asphaltic Bitumen; Hot Process," British Standard Spec. No. 594–1935, British Standards Institution; "Rolled Asphalt; Fluxed Natural Asphalt and Asphaltic Bitumen; Hot Process," British Standard Spec. No. 595–1935, British Standards Institution; "Compressed Rock Asphalt," British Standard Spec. No. 348–1935, British Standards Institution.

97 [p. 592] **U. S. Pat.** 607884 of Jul. 26, 1898 to Clifford Richardson.

98 [p. 592] "The Calculation of the Required Binding Material in the Preparation of Asphalt and Tar Coverings," by R. Wilhelmi, *Erdöl und Teer*, **6**, 481 (1930); "Die Kornumbildung bei der Verdichtung als Hinweis auf den richtigen Kornaufbau bituminöser Deckenbeläge," by R. Wilhelmi, *Bitumen*, **5**, 177 (1935).

99 [p. 592] "The Effect of the Mixing Temperature on the Physical Properties of an Oil Asphalt in a Sheet Asphalt Mixture," by J. H. Bateman and H. L. Lehmann, *Proc. A.S.T.M.*, **29**, II, 943 (1929).

100 [p. 593] Private communication from T. G. Marriott, London.

101 [p. 593] **U. S. Pat.** 1812730 of Jun. 30, 1931 to W. C. West.

102 [p. 594] **U. S. Pat.** 743020 of Nov. 3, 1903 to J. W. Nesmith; **Ger. Pat.** 90923 of Jul. 9, 1895 to United States Repair and Guaranty Co.

103 [p. 594] **Ger. Pat.** 96864 of Jan. 27, 1897 to J. W. Buzzard.

104 [p. 594] **French Pat.** 758470 of Jan. 18, 1934 to C. G. J. Lefebvre.

105 [p. 594] **U. S. Pat.** 938698 of Nov. 2, 1909 to J. A. W. Pine; **Can. Pat.** 345946 of Nov. 13, 1934 to N. H. Taylor; **Brit. Pat.** 393454 of Sep. 27, 1932 to N. H. Taylor; 456922 of Jul. 18, 1935 to N. H. Taylor.

106 [p. 594] "Federal Spec. for Sand; (for) Grout-filler, Brick and Stone-Block Pavements," Federal Standard Stock Catalogue, Section IV, (Part 5), SS–S–61 (Jun. 23, 1931).

107 [p. 596] "Asphalt Block Pavements," Brochure No. 15, 2nd Edition Revised, The Asphalt Association, New York (1926).

108 [p. 598] "Standard Definitions of Processes (for Wood Preservation)," Spec. No. 12, adopted by the American Wood-Preservers' Association, Chicago, Ill. (1921); "Standard Specification for Creosoted Wood-block Paving," Spec. No. 16, amended and adopted by the American Wood-Preservers' Association, Chicago, Ill., (1923); "Standard Specification for Interior Creosoted Wood-block Flooring," Spec. No. 19, adopted by the American Wood-

Preservers' Association, Chicago, Ill., (1923); "Standard Specifications for Wooden Paving Blocks for Exposed Pavements," (D 52–20), A.S.T.M. Standards 1936, II, 462.

109 [p. 599] **U. S. Pat.** 1448614 of Mar. 13, 1923 to A. H. Carrier.

110 [p. 600] "Standard Definitions of Terms Relating to Timber Preservatives," (D 324–33), A.S.T.M. Standards 1936, II, 564; "Standard Volume and Specific Gravity Correction Tables for Creosote, Creosote-coal-tar Solution (up to 50 per cent Tar) and Coal Tar (Coke-oven Tars)," (D 347–33), A.S.T.M. Standards 1936, II, 556.

111 [p. 600] Specifications adopted by the American Wood-Preservers' Association, Chicago, Ill.; A, B and C represent Spec. No. 15 adopted 1923; D, E and F represent Spec. No. 4 adopted 1921, 1923 and 1924 respectively; G represents Spec. No. 5 adopted 1923; H represents Spec. No. 31 adopted 1924; and I represents Spec. No. 32 adopted 1924; "Coal-tar and Water-gas Creosotes—Their Properties and Methods of Testing," by Ernst Bateman, Bull. No. 1036, U. S. Dept. of Agriculture, Wash., D. C. (1922); "Standard Specifications for Creosote," (D 390–36), A.S.T.M. Standards 1936, II, 527; "Standard Specifications for Creosote Coal-tar Solution," (D 391–36), A.S.T.M. Standards 1936, II, 529; "Federal Spec. for Wood-Preservative; Creosote (for) Brush and Spray Treatment," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-W-561a (Jan. 9, 1934); "Federal Spec. for Wood-Preservative; Coal-tar Creosote (for) Ties and Structural Timbers," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-W-556, (Apr. 28, 1931); "Federal Spec. for Wood-Preservative; Creosote-Coal-Tar-Solution (for) Ties and Structural Timbers," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-W-566, (Apr. 28, 1931); "Federal Spec. for Wood-Preservative; Preservative Treatment," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-W-571a. (Jan. 9, 1934); "Creosote for the Preservation of Timber," British Standard Spec. No. 144–1921 (Add. Nov. 1928), British Standards Institution.

112 [p. 602] **Ger. Pat.** 100979 of Dec. 11, 1897 to P. Decauville.

113 [p. 602] **U. S. Pat.** 1988178 of Jan. 15, 1935 to D. R. Merrill.

114 [p. 602] **Brit. Pat.** 249698 of May 25, 1925 to P. R. Chambige.

115 [p. 602] "Sand for Cement-Mortar Bed for Brick, Stone-block, or Wood-block Pavements," Federal Spec. Board Spec. No. 462; "Federal Spec. for Coarse-Aggregate; (for) Portland Cement-Concrete-Pavement or Base (Crushed Stone, Gravel, or Slag)," Federa Standard Stock Catalogue, Section IV, (Part 5), SS-C-571, (Feb. 3, 1931).

116 [p. 602] "Federal Spec. for Materials; (for) Cushion Course, Brick, Stone-Block or Wood-Block Pavements (Sand, Slag, Limestone Screenings, etc.)," Federal Standarc Stock Catalogue, Section IV, (Part 5), SS-M-51, (Jun. 23, 1931).

117 [p. 602] "Standard Specifications for Materials for Cement Grout Filler for Brick and Stone Block Pavements," (D 57–20), A.S.T.M. Standards 1936, II, 1013; Americar Tentative Standard A.S.A. No.: A-31-1924; "Standard Specifications for Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements," (D 58–24), A.S.T.M. Standards 1936, II, 1015; "Federal Spec. for Sand; (for) Cement-Mortar-Bed, Brick, Stone-Block, or Wood-Block Pavements," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-S-51, (Jan. 25, 1927).

118 [p. 603] **Ger. Pat.** 14762 of Feb. 23, 1881 to L. von Hegnenberg.

119 [p. 603] "Standard Specification for Creosote Wood-block Street Paving," Spec. No. 16, adopted by the American Wood-Preservers' Association, Chicago, Ill., (1923); "Federal Spec. for Tar; (for) Joint-Filler," Federal Standard Stock Catalogue, Section IV, (Part 5), R-T-111, (Feb. 7, 1933).

120 [p. 604] British Standard Spec. Nos. 596 and 597, "Mastic Asphalt Surfacing," British Standards Institution, London (1935).

121 [p. 604] " The Principles of the Manufacture of Mastic Asphalt," by A. W. Attwooll, *J. Soc. Chem. Ind.*, **49**, 181T (1930); "Experience with some Applications of Mastic Asphalt in a Chemical Works," by Donald McDonald, *J. Soc. Chem. Ind.*, **49**, 187T (1930).

122 [p. 605] **Can. Pat.** 140182 of May 7, 1912 to H. W. Johns-Manville Co.

123 [p. 605] **U. S. Pat.** 1740212 of Dec. 17, 1929 to G. C. Warren.

124 [p. 605] "Standard Specifications for Asphalt Mastic for Use in Waterproofing," (D 169–25), A.S.T.M. Standards 1936, II, 1125; "Standard Specifications for Acid-Resist-

ing Asphalt Mastic," (D 223–30), A.S.T.M. Standards 1936, II, 1123; "Mastic Asphalt Surfacing; Fluxed Lake Asphalt and Asphaltic Bitumen; Hot Process," British Standard Spec. No. 596–1935, British Standards Institution; "Mastic Asphalt Surfacing; Fluxed Natural Asphalt and Asphaltic Bitumen; Hot Processs," British Standard Spec. No. 597–1935; British Standards Institution.

125 [p. 606] **U. S. Pat.** 1305678 of Jun. 3, 1919 to Henry Wiederhold; **Brit. Pats.** of 1890 (Aug. 29), 13595 to H. P. Williams and E. G. Williams; 151639 of Sep. 27, 1920 to Barber Asphalt Paving Co.; 398268 of Mar. 19, 1932 to E. W. Harvey and H. F. Weatherby.

126 [p. 606] **Ger. Pats.** 321029 of Oct. 13, 1917 to J. Frenz; 329824 of Feb. 26, 1919 to J. Frenz.

127 [p. 606] **Brit. Pat.** 373994 of Nov. 29, 1930 to W. E. Smith.

128 [p. 607] **Can. Pat.** 346113 of Nov. 20, 1934 to C. R. Norman.

129 [p. 607] **Ger. Pat.** 63437 of Nov. 21, 1891 to Ludwig Haarmann.

130 [p. 608] **U. S. Pat.** 1193843 of Aug. 8, 1916 to A. E. Symmes.

131 [p. 608] **U. S. Pat.** 1854435 of Apr. 19, 1932 to L. S. Wilbur.

132 [p. 608] **U. S. Pat.** 1824418 of Sep. 22, 1931 to L. S. Wilbur.

133 [p. 608] **U. S. Pat.** 1754537 of Apr. 15, 1930 to L. S. Wilbur.

134 [p. 609] **U. S. Pat.** 1688041 of Oct. 16, 1928 to C. N. Forrest, J. S. Miller, Jr. and C. S. Babb.

135 [p. 609] **Ger. Pats.** 4993 of Sep. 7, 1878 to J. Brandt; 58087 of Nov. 23, 1890 to Ch. Claussen; 87704 of Jun. 16, 1895 to Battista Bianchi; 119866 of Feb. 25, 1899 to Arthur Metz.

136 [p. 609] **Ger. Pat.** 11498 of Mar. 3, 1880 to A. Riebeck.

137 [p. 609] Private communication from T. G. Marriott, London.

138 [p. 611] "Asphalt Mastic Roofing," by D. M. Wilson, *Chemistry & Industry*, **52**, 959 (1933).

139 [p. 611] **U. S. Pats.** 547097 of Oct. 1, 1895 to Henry Van Newkirk; 547104 of Oct. 1, 1895 to J. C. Zallée.

140 [p. 611] **Brit. Pat.** 418635 of Apr. 8, 1933 to A. S. H. Berdeaux.

141 [p. 611] **Ger. Pat.** 23440 of Nov. 11, 1882 to Carl Wildhagen.

142 [p. 612] **U. S. Pats.** 1134939 of Apr. 6, 1915 to James Banwell; 1803178 of Apr. 28, 1931 to A. C. Fischer; **Can. Pats.** 327610 and 327611 of Nov. 15, 1932 to Barrett Co.

143 [p. 612] **U. S. Pats.** 1785457 of Dec. 16, 1930 to A. J. Pfohl; 1881440 of Oct. 11, 1932 to A. C. Fischer.

144 [p. 612] **U. S. Pat.** 1826985 of Oct. 13, 1931 to A. E. Brown.

145 [p. 612] **U. S. Pat.** 1207524 of Dec. 5, 1916 to C. N. Forrest.

146 [p. 612] **Can. Pat.** 338880 of Jan. 23, 1934 to Philip Carey Mfg. Co.

147 [p. 612] **U. S. Pat.** 1827193 of Oct. 13, 1931 to A. C. Fischer, B. A. Stewart and H. W. Herbst.

148 [p. 613] **U. S. Pats.** 1156122 of Oct. 12, 1915 to J. C. Woodley; 1166166 of Dec. 28, 1915 to J. C. Woodley; 1177267 of Mar. 28, 1916 to R. P. Perry; 1240524 of Sep. 18, 1917 to J. C. Woodley.

149 [p. 613] **U. S. Pats.** 226547 of Apr. 13, 1880 to J. L. Pope; 1680144 of Aug. 7, 1928 to A. C. Fischer; 1691234 of Nov. 13, 1928 to A. C. Fischer; 1890260 of Dec. 6, 1932 to J. A. MacDonald; 1892659 of Dec. 27, 1932 to W. R. Meadows; **Brit. Pats.** 277513 of Oct. 9, 1926 to D. L. Irwin; 427851 of Jun. 13, 1934 to Jacques Gevers; 428149 of Nov. 11, 1933 to W. L. Miller.

150 [p. 613] **U. S. Pats.** 1571874 of Feb. 2, 1926 to A. C. Fischer; 1694212 of Dec. 4, 1928 to A. C. Fischer; 1770356 of Jul. 8, 1930 to A. C. Fischer; 1807836 of Jun. 2, 1931 to A. C. Fischer; 1911931 of May 30, 1933 to Albert Skinner; 2019449 of Oct. 29, 1935 to A. C. Fischer.

151 [p. 613] **U. S. Pats.** 1770358 of Jul. 8, 1930 to A. C. Fischer; 1814139 of Jul. 14, 1931 to A. C. Fischer.

152 [p. 613] **U. S. Pat.** 1909202 of May 16, 1933 to C. F. Lytle.

153 [p. 613] **U. S. Pats.** 63371 of Apr. 2, 1867 to D. W. Denton; 1582880 of Apr. 27,

1926 to A. C. Fischer; 1680144 of Aug. 7, 1928 to A. C. Fischer; 1689945 of Oct. 30, 1928 to A. C. Fischer; 1726612 and 1726613 of Sep. 3, 1929 to A. C. Fischer; 1807905 of Jun. 2, 1931 to A. C. Fischer; 1839181 of Dec. 29, 1931 to A. C. Fischer; Reissue 18642 of Nov. 1, 1932 to A. C. Fischer.

154 [p. 613] **U. S. Pats.** 1683881 of Sep. 11, 1928 to A. C. Fischer; 1689945 of Oct. 30, 1928 to A. C. Fischer; 1744303 of Jan. 21, 1930 to A . C. Fischer.

155 [p. 613] **U. S. Pats.** 1597273 of Aug. 24, 1926 to A. C. Fischer; 1680144 of Aug. 7, 1928 to A. C. Fischer.

156 [p. 613] **U. S. Pat.** 1769208 of Jul. 1, 1930 to T. C. Crowley.

157 [p. 613] **U. S. Pats.** 855792 of Jun. 4, 1907 to J. E. Lappen; 1680144 of Aug. 7, 1928 to A. C. Fischer; 1820217 of Aug. 25, 1931 to A. C. Fischer.

158 [p. 613] **U. S. Pat.** 1730000 of Oct. 1, 1929 to A. C. Fischer.

159 [p. 613] **U. S. Pat.** 1911140 of May 23, 1933 to A. C. Fischer.

160 [p. 613] **U. S. Pats.** 1597273 of Aug. 24, 1926 to A. C. Fischer; 1728114 of Sep. 10, 1929 to A. C. Fischer; 1742650 and 1742651 of Jan. 7, 1930 to A. C. Fischer; 1748016 of Feb. 18, 1930 to A. C. Fischer; 1770360 of Jul. 8, 1930 to A. C. Fischer; 1793439 of Feb. 17, 1931 to A. C. Fischer; 1803178 of Apr. 28, 1931 to A. C. Fischer; 1809800 and 1809801 of Jun. 9, 1931 to A. C. Fischer; 1817933 of Aug. 11, 1931 to E. S. Ross; 1844467 of Feb. 9, 1932 to A. C. Fischer; 1882792 of Oct. 18, 1932 to A. C. Fischer; 1901870 of Mar. 21, 1933 to A. C. Fischer; 1911139 of May 23, 1933 to A. C. Fischer; 1952706 of Mar. 27, 1934 to A. C. Fischer; 2016404 of Oct. 8, 1935 to L. E. Warner; 2023529 of Dec. 10, 1935 to H. C. Jussen; 2040256 of May 12, 1936 to F. R. Grant; Reissue 20029 of Jul. 7, 1936 to A. C. Fischer; 2053281 of Sep. 8, 1936 to G. B. Gaiennie; **Can. Pats.** 258163 of Feb. 16, 1926 to G. C. Warren; 328056 of Nov. 29, 1932 to Philip Carey Mfg. Co.

161 [p. 613] **U. S. Pat.** 1899435 of Feb. 28, 1933 to A. C. Fischer.

162 [p. 613] **U. S. Pats.** 1689945 of Oct. 30, 1928 to A. C. Fischer; 1728114 of Sep. 10, 1929 to A. C. Fischer; 1770356 of Jul. 8, 1930 to A. C. Fischer; 1793439 of Feb. 17, 1931 to A. C. Fischer.

163 [p. 613] **U. S. Pats.** 1680144 of Aug. 7, 1928 to A. C. Fischer; 1743765 of Jan. 14, 1930 to A. C. Fischer.

164 [p. 613] **U. S. Pats.** 1503942 of Aug. 5, 1924 to A. C. Fischer; Reissue 16554 of Feb. 22, 1927 to A. C. Fischer; 1680144 of Aug. 7, 1928 to A. C. Fischer; 1728115 of Sep. 10, 1929 to A. C. Fischer; 1751196 of Mar. 18, 1930 to A. C. Fischer.

165 [p. 613] **U. S. Pat.** 1881436 of Oct. 11, 1932 to A. C. Fischer.

166 [p. 613] **Can. Pat.** 335953 of Sep. 26, 1933 to Johns-Manville Corp.

167 [p. 613] **U. S. Pat.** 1965403 of Jul. 3, 1934 to G. H. Alvey.

168 [p. 613] **U. S. Pat.** 1591968 of Jul. 13, 1926 to A. C. Fischer.

169 [p. 613] **U. S. Pat.** 1862506 of Jun. 7, 1932 to A. C. Fischer.

170 [p. 613] **U. S. Pat.** 1769629 of Jul. 1, 1930 to A. C. Fischer.

171 [p. 613] **U. S. Pats.** 1951728 of Mar. 20, 1934 to A. C. Fischer; 1982506 of Nov. 27, 1934 to A. C. Fischer; 1987530 of Jan. 8, 1935 to J. S. Hipple; 1987553 of Jan. 8, 1935 to A. C. Fischer.

172 [p. 613] **U. S. Pats.** 1591670 of Jul. 6, 1926 to A. C. Fischer; Reissue 17369 of Jul. 9, 1929 to A. C. Fischer; 1744378 of Jan. 21, 1930 to A. C. Fischer; 1838036 of Dec. 22, 1931 to A. C. Fischer; 2065521 of Dec. 29, 1936 to A. C. Fischer.

173 [p. 613] **U. S. Pats.** 1665718 of Apr. 10, 1928 to C. J. Pater; 1706110 of Mar. 19, 1929 to A. C. Fischer; Reissue 17792 of Sep. 2, 1930 to C. J. Pater; 1961847 of Jun. 5, 1934 to A. C. Fischer.

174 [p. 613] **U. S. Pat.** 1769629 of Jul. 1, 1930 to A. C. Fischer.

175 [p. 613] **U. S. Pats.** 1755107 of Apr. 15, 1930 to A. C. Fischer; 1770167 of Jul. 8, 1930 to A. C. Fischer; 1770357 of Jul. 8, 1930 to A. C. Fischer; 1770361 of Jul. 8, 1930 to A. C. Fischer; 1821885 of Sep. 1, 1931 to A. C. Fischer; Reissue 18765 of Mar. 14, 1933 to A. C. Fischer; Reissue 19183 of May 29, 1934 to A. C. Fischer.

176 [p. 613] **U. S. Pats.** 1863104 of Jun. 14, 1932 to A. C. Fischer; 2067707 of Jan. 12, 1937 to A. C. Fischer; **Can. Pat.** 328055 of Nov. 29, 1932 to Philip Carey Mfg. Co.

177 [p. 613] **U. S. Pat.** 1769635 of Jul. 1, 1930 to A. C. Fischer.

178 [p. 613] **U. S. Pat.** 1639764 of Aug. 23, 1927 to A. C. Fischer.

179 [p. 613] **U. S. Pats.** 1800193 and 1800194 of Apr. 7, 1931 to A. C. Fischer.

180 [p. 614] **U. S. Pats.** 1328107 of Jan. 13, 1920 to S. B. Waring; 2072105 of Mar. 2, 1937 to A. C. Fischer; **Brit. Pat.** of 1894 (Aug. 18), 15756 to R. J. Goddard.

181 [p. 614] **U. S. Pats.** 1040093 of Oct. 1, 1912 to James Adkins, Jr.; 1220766 of Mar. 27, 1917 to W. J. Moeller; 1280572 of Oct. 1, 1918 to A. S. Spiegel; 1339094 of May 4, 1920 to F. C. Bowman; Reissue 15840 of May 13, 1924 to H. C. Riehle; 1566319 of Dec. 22, 1925 to A. C. Fischer; 1570970 of Jan. 26, 1926 to J. J. Milar; 1572261 of Feb. 9, 1926 to E. J. Yetter; 1572474 of Feb. 9, 1926 to A. C. Fischer; 1731334 and 1731336 of Oct. 15, 1929 to A. C. Fischer; 1769630 of Jul. 1, 1930 to A. C. Fischer; 1881439 of Oct. 11, 1932 to A. C. Fischer; 2012370 of Aug. 27, 1935 to James Banwell.

182 [p. 614] **U. S. Pats.** 635176 of Oct. 17, 1899 to T. K. Miner; 1248909 of Dec. 4, 1917 to H. B. Pullar.

183 [p. 614] **U. S. Pat.** 1248909 of Dec. 4, 1917 to H. B. Pullar.

184 [p. 614] **U. S. Pats.** 1582638 of Apr. 27, 1926 to R. B. Crabbs; 1730066 of Oct. 1, 1929 to A. C. Fischer.

185 [p. 614] **U. S. Pats.** 1622311 of Mar. 29, 1927 to A. C. Fischer; 1701629 of Feb. 12, 1929 to H. B. Pullar; 1881437 of Oct. 11, 1932 to A. C. Fischer.

186 [p. 614] **U. S. Pat.** 105599 of Jul. 19, 1870 to J. J. Schillinger.

187 [p. 614] **U. S. Pats.** 1737318 of Nov. 26, 1929 to R. E. Martin; 1818874 of Aug. 11, 1931 to G. J. Ulrich; **Can. Pat.** 315117 of Sep. 8, 1931 to C. E. Ulrich.

188 [p. 614] **U. S. Pat.** 635176 of Oct. 17, 1899 to T. K. Muir.

189 [p. 614] **U. S. Pat.** 2005139 of Jun. 18, 1935 to A. C. Fischer.

190 [p. 614] **U. S. Pats.** 1220766 of Mar. 27, 1917 to W. J. Moeller; Reissue 14871 of Jun. 1, 1920 to W. J. Moeller; 1239573 of Sep. 11, 1927 to J. A. de Mond; 1730066 of Oct. 1, 1929 to A. C. Fischer; 1841039 of Jan. 12, 1932 to W. F. Kleeman; 1846325 of Feb. 23, 1932 to A. C. Fischer; 1913235 of Jun. 6, 1933 to N. P. Harshberger; **Brit. Pat.** 342296 of Oct. 25, 1929 to H. D. Elkington.

191 [p. 614] **U. S. Pat.** 1961077 of May 29, 1934 to C. J. Pater.

192 [p. 614] **U. S. Pats.** 1662567 of Mar. 13, 1928 to A. C. Fischer; 1769630 of Jul. 1, 1930 to A. C. Fischer.

193 [p. 615] **U. S. Pats.** 1603506 of Oct. 19, 1926 to E. L. Benedict and R. L. Glose; Reissue 18188 of Sep. 15, 1931 to E. L. Benedict and R. L. Glose; 1907416 of May 2, 1933 to A. C. Fischer.

194 [p. 615] **U. S. Pats.** 1558799 of Oct. 27, 1925 to A. C. Fischer; 1701628 of Feb. 12, 1929 to H. B. Pullar.

195 [p. 615] **U. S. Pats.** 1329631 of Feb. 3, 1920 to H. C. Riehle; Reissue 15840 of May 13, 1924 to H. C. Riehle.

196 [p. 615] **U. S. Pats.** 1195368 of Aug. 22, 1916 to E. M. Laing; 1241405 of Sep. 25, 1917 to W. E. Leach; 1298018 of Mar. 25, 1919 to A. E. Davis; 1588718 of Jun. 15, 1926 to A. C. Fischer; 1655835 of Jan. 10, 1928 to A. C. Fischer; 1694213 of Dec. 4, 1928 to A. C. Fischer; 1730067 of Oct. 1, 1929 to A. C. Fischer; 1743766 of Jan. 14, 1930 to A. C. Fischer; 1762682 of Jun. 10, 1930 to A. C. Fischer; 1997546 of Apr. 9, 1935 to A. C. Fischer; **Can. Pat.** 264139 of Sep. 7, 1926 to E. J. Yetter.

197 [p. 615] **U. S. Pats.** 1078982 of Nov. 18, 1913 to James Banwell; 1089943 of Mar. 10, 1914 to S. T. Morse and W. D. P. Warren; 1701628 of Feb. 12, 1929 to H. B. Pullar; 1769989 and 1769990 of Jul. 8, 1930 to A. C. Fischer; 1770168 of Jul. 8, 1930 to A. C. Fischer; 1998421 of Apr. 16, 1935 to A. C. Fischer; **Can. Pat.** 157422 of Aug. 18, 1914 to James Banwell.

198 [p. 615] **U. S. Pat.** 1903756 of Apr. 18, 1933 to A. C. Fischer.

199 [p. 615] **U. S. Pat.** 1085275 of Jan. 27, 1914 to W. P. Lonsdale.

200 [p. 615] **U. S. Pat.** 1223045 of Apr. 17, 1917 to L. R. Ferguson.

201 [p. 615] **U. S. Pat.** 1637178 of Jul. 26, 1927 to A. C. Fischer; **Brit. Pat.** 357693 of Jul. 24, 1930 to N. E. Snow.

202 [p. 615] **U. S. Pats.** 1639763 of Aug. 23, 1927 to A. C. Fischer; 1735270 of Nov. 12, 1929 to A. C. Fischer.

203 [p. 615] **U. S. Pat.** 1898943 of Feb. 21, 1933 to A. C. Fischer.

204 [p. 615] **U. S. Pats.** 1840085 and 1840086 of Jan. 5, 1932 to A. C. Fischer.

205 [p. 615] **U. S. Pats.** 1609077 of Nov. 30, 1926 to A. C. Fischer; 1612274 of Dec. 28, 1926 to A. C. Fischer; 1619924 of Mar. 8, 1927 to A. C. Fischer; 1651879 of Dec. 6, 1927 to A. C. Fischer; 1689278 of Oct. 30, 1928 to G. D. Crabbs; 1689300 of Oct. 30, 1928 to H. L. Rogers; 1730068 of Oct. 1, 1929 to A. C. Fischer; 1744304 of Jan. 21, 1930 to A. C. Fischer; 1744714 of Jan. 21, 1930 to A. C. Fischer; 1755108 of Apr. 15, 1930 to A. C. Fischer; 1803179 of Apr. 28, 1931 to A. C. Fischer; 1808302 of Jun. 2, 1931 to A. C. Fischer; 1813199 and 1813200 of Jul. 7, 1931 to H. L. Rogers; 1881660 of Oct. 11, 1932 to Eugene Keller, Jr.; **Ger. Pats.** 3204 of May 2, 1878 to C. F. Werckner; 3575 of May 7, 1878 to J. Brandt & G. W. von Navrocki; 85826 of Mar. 27, 1895 to Otto Storsberg and Carl Weigandt.

206 [p. 616] **U. S. Pats.** 1941435 of Dec. 26, 1933 to A. C. Fischer; 1952219 of Mar. 27, 1934 to A. J. Pfohl; 1970037 of Aug. 14, 1934 to A. C. Fischer; 2051532 of Aug. 18, 1936 to H. L. Rogers; **Can. Pat.** 363491 of Jan. 19, 1937 to Philip Carey Mfg. Co.

207 [p. 617] "Der Einbau von Asphaltdecken auf Böschungen," by Fr. Joedicke, *Bitumen*, 6, 163 (1936); **U. S. Pats.** 1987151 of Jan. 8, 1935 to L. R. Mason; 1991196 of Feb. 12, 1935 to A. C. Deer; 2001130 of May 14, 1935 to J. R. Fordyce; 2047197 of Jul. 14, 1936 to J. R. Fordyce; 2051578 of Aug. 18, 1936 to W. L. Schloss and E. G. Bell; **Ger. Pat.** Appl. B–166704 of Sep. 1, 1934 to Baeumer & Loesch.

CHAPTER XXIX

1 [p. 617] "Methods in Refining Roofing Asphalts," by W. W. Peters, *Oil and Gas J.*, **27** (42), 113 and 163 (1929); "Vom Pappdach," by Direktor Ing. V. D. I.-Belani, *Asphalt u. Teer*, **17**, 472 (1930); "Das deutsche Dach im alten Griechenland und in Italien," by K. Stuhl, Deutsches Dachdecker-Handwerk, **51**, 812 and 855 (1930); "Asphalt Shingles," by by J. M. Weiss, *J. Ind. Eng. Chem.*, **23**, 168 (1931).

2 [p. 618] "Classification of Rag Stock," issued by the Felt Manufacturers' Association of the U. S. (Feb. 16, 1928); "Experimental Production of Roofing Felts," by M. B. Shaw, G. W. Bicking and O. G. Strieter, Research Paper No. 67, Bureau of Standards, Washington, D. C. (Jun. 1929); "A Study of the Weathering Quality of Roofing Felts Made from Various Fibers," by O. G. Strieter, Research Paper RP–888, National Bureau of Standards, U. S. Dept. of Commerce (May 1936); *J. of Research*, National Bureau of Standards, **16** (May 1936).

3 [p. 621] "Relative Permanence of Papers Exposed to Sunlight," by G. A. Richter, *J. Ind. Eng. Chem.*, **27**, 177 (1935).

4 [p. 621] **U. S. Pats.** 119601 of Oct. 3, 1871 and 121166 of Nov. 21, 1871 to J. K. Griffin; 253656 of Feb. 14, 1882 to S. M. Allen; 1188495 of Jun. 27, 1916 to E. J. Schroder; 1365878 of Jan. 18, 1921 to H. F. Weiss; 1376285 of Apr. 26, 1921 to Rudolf Kron; **Brit. Pat.** 135466 of Nov. 19, 1918 to Rudolf Kron.

5 [p. 621] **U. S. Pat.** 1948979 of Feb. 27, 1934 to P. W. Codwise; **Can. Pat.** 361273 of Oct. 20, 1936 to Certainteed Products Co.

6 [p. 621] **U. S. Pat.** 1467594 of Sep. 11, 1923 to H. F. Weiss.

7 [p. 621] **U. S. Pats.** 1639704 of Aug. 23, 1927 to G. A. Richter and M. O. Schur; 1700596 of Jan. 29, 1929 to G. A. Richter; 1709322 of Apr. 16, 1929 to G. A. Richter; 1742219 of Jan. 7, 1930 to G. A. Richter; 1773320 of Aug. 19, 1930 to G. A. Richter; 1784161 of Dec. 9, 1930 to G. A. Richter and M. O. Schur; 1790839 of Feb. 3, 1931 to G. A. Richter; 1801782 of Apr. 21, 1931 to G. A. Richter and M. O. Schur; 1802575 of Apr. 28, 1931 to G. A. Richter; 1802984 of Apr. 28, 1931 to G. A. Richter; 1809287 of Jun. 9, 1931 to G. A. Richter; 1829157 of Oct. 27, 1931 to G. A. Richter; 1833976 of Dec. 1, 1931 to G. A. Richter and M. O. Schur; 1838326 of Dec. 29, 1931 to G. A. Richter; 1857432 and 1857433 of May 10, 1932 to P. W.

Codwise; 1888771 and 1888772 of Nov. 22, 1932 to G. A. Richter and H. A. Chase; 1947103, 1947104, 1947105, 1947106, 1947107 and 1947108 of Feb. 13, 1934 to J. E. Plumstead; 1956045 of Apr. 24, 1934 to G. A. Richter; 1958976 of May 15, 1934 to G. A. Richter; 1961945 and 1961946 of Jun. 5, 1934 to M. O. Schur and W. L. Hearn; 1972055 of Aug. 28, 1934 to G. A. Richter; 2033485 of Mar. 10, 1936 to M. O. Schur and W. L. Hearn; 2047978 of Aug. 4, 1936 to M. O. Schur and W. L. Hearn; 2051168 of Aug. 18, 1936 to H. W. Greider and G. A. Fasold. **Can. Pat.** 335906 of Sep. 26, 1933 to Brown Co.; 343125 of Jul. 17, 1934 to Brown Co.; 345960 of Nov. 13, 1934 to Brown Co.; 340392 and 340393 of Mar. 27, 1934 to Brown Co.; 353556 and 353557 of Oct. 15, 1935 to Brown Co.

8 [p. 622] **U. S. Pats.** 1305770 of Jun. 3, 1919 to A. L. Clapp; 1365878 of Jan. 18, 1921 to H. F. Weiss; 1372640 of Mar. 22, 1921 and 1374187 of Apr. 12, 1921 to A. L. Clapp; 2057166 of Oct. 13, 1936 to M. O. Schur; **Can. Pats.** 47031 of Sep. 12, 1894 to C. H. Dalrymple; 56014 of May 21, 1897 to William Angus; 352718 of Sep. 3, 1935 to Philip Carey Mfg. Co.

9 [p. 622] **Can. Pat.** 285687 of Dec. 18, 1928 to Barrett Co.

10 [p. 622] **U. S. Pats.** 1310376 of Jul. 15, 1919 to A. W. Schorger; 1376285 of Apr. 26, 1921 to Rudolf Kron; **Brit. Pat.** 135466 of Nov. 19, 1918 to Rudolf Kron.

11 [p. 622] **Can. Pat.** 285688 of Dec. 18, 1928 to Beaver Co., Ltd.

12 [p. 622] "The Use of Bark for Paper Specialties," by Otto Kress, *J. Ind. Eng. Chem.*, **8**, 883 (1916). **U. S. Pats.** 253656 of Feb. 14, 1882 to S. M. Allen; 311938 and 311939 of Feb. 10, 1885 to C. W. Armour; 1263823 of Apr. 23, 1918 to V. A. Wallin and O. A. Heppes; 1362850 of Dec. 21, 1920 to A. L. Clapp; 1797901 of Mar. 24, 1931 to E. R. Darling; 1979864 of Nov. 6, 1934 to F. L. Carson; **Can. Pats.** 25512 of Dec. 6, 1886 to J. M. Allen; 25584 of Dec. 18, 1886 to J. M. Allen; 29558 of Jul. 25, 1888 to T. P. Bishop; **Brit. Pats.** of 1801 (Jun. 2), 2507 to Thomas Winter; of 1861 (Feb. 2), 282 to W. Clark; 177834 of Apr. 3, 1922 to C. C. Muench; **Jap. Pat.** 35965 of Mar. 11, 1920 to Y. Sakakibara.

13 [p. 622] **U. S. Pats.** 311938 and 311939 of Feb. 10, 1885 to C. W. Armour; 854740 of May 28, 1907 to A. G. Hennion; 1924815 of Aug. 29, 1933 to Max Skolnik; 2029310 of Feb. 4, 1936 to Pierre Drewsen; 2037268 of Apr. 14, 1936 to C. S. Reeve; 2037297 of Apr. 14, 1936 to F. W. Yeager; **Can. Pat.** 301852 of Jul. 8, 1930 to Barrett Co.

14 [p. 622] **U. S. Pats.** 63087 of Mar. 19, 1867 and 71210 of Nov. 19, 1867 to Antonio Pelletier; 88516 of Mar. 30, 1869 to R. W. Russell; 130615 of Aug. 20, 1872 to W. P. Arnold.

15 [p. 622] **U. S. Pats.** 1237000 of Aug. 14, 1917 and 1326232 of Dec. 30, 1919 to Herman von Uffel; **Brit. Pats.** of 1857 (Mar. 21), 806 to E. Hyde; of 1863 (Jun. 8), 1423 to Henry Reynell; of 1865 (Feb. 18), 465 to Christopher Brakell, William Hoehl and William Günther.

16 [p. 622] **U. S. Pat.** 1184112 of May 23, 1916 to W. A. R. M. McRae.

17 [p. 622] **U. S. Pats.** 1139305 of May 11, 1915 and 1146189 and 1146190 of Jul. 13, 1915 to H. B. McFarland and R. J. Shoemaker; 1226738 of May 22, 1917 to Herbert Abraham; 1252468 of Jan. 8, 1918 to H. B. McFarland and R. J. Shoemaker; 1311941 of Aug. 5, 1919 to Herbert Abraham; **Can. Pat.** 178318 of Jul. 24, 1917 to Herbert Abraham. **Brit. Pats.** of 1861 (Jan. 5), 28, to P. Courtais and F. Jammet; of 1862 (Aug. 27), 3272 to H. Harben; of 1864 (Apr. 28), 1072 to T. G. Ghislin.

18 [p. 622] **U. S. Pats.** 1305770 of Jun. 3, 1919 to A. L. Clapp; 1328267 of Jan. 20, 1920 to C. P. Cowan; **Brit. Pat.** of 1881 (Jul. 28), 3297 to H. Armstrong and J. A. London.

19 [p. 622] "Beiträge zur Kenntnis der Kapokfaser," by C. G. Schwalbe and R. Troeltzsch, Der Tropenpflanzer, **17**, (No. 12), 655–660 (1913); **U. S. Pat.** 1450600 of Apr. 3, 1923 to August Lendle; 1973722 of Sep. 18, 1934 to W. C. Nash; 2036195 of Apr. 7, 1936 to J. T. Castles, Jr.; **Can. Pat.** 350922 of Jun. 11, 1935 to Seaman Paper Co.; **Brit. Pat.** 449979 of Jun. 26, 1935 to F. P. Dehn.

20 [p. 622] **U. S. Pat.** 2013841 of Sep. 10, 1935 to G. A. Richter.

21 [p. 622] **U. S. Pat.** 1850791 of Mar. 22, 1932 to Pierre Drewsen.

22 [p. 622] **U. S. Pats.** 63087 of Mar. 19, 1867 and 71210 of Nov. 19, 1867 to Antonio Pelletier; 1229422 of June 12, 1917 to Viggo Drewsen; 1599253 of Sep. 7, 1926 to Max Skolnik; **Can. Pat.** 293510 of Oct. 1, 1929 to Max Skolnik.

23 [p. 622] **Can. Pat.** 285686 of Dec. 18, 1928 to Barrett Co.

24 [p. 622] "Production of Roofing Carton from Reed." by N. N. Orlov and V. F.

Osmulski, Bumazh. *Prom.*, 13, 60, (1934); **U. S. Pat.** 2041745 of May 26, 1936 to Henry Dreyfus.

25 [p. 622] **U. S. Pat.** 1389936 of Sep. 6, 1921 to A. L. Clapp.

26 [p. 622] **U. S. Pats.** 40592 of Nov. 17, 1863 to S. M. Allen; 303301 of Aug. 12, 1884 to Carl Lortzing; 743031 of Nov. 3, 1903 to G. T. Pratt; 1021660 of Mar. 26, 1912 to A. L. Clapp; 1211837 of Jan. 9, 1917 to C. N. Forrest; 1234315 of Jul. 24, 1917 to E. B. Eising; 1269905 of Jun. 18, 1918, 1305770 of Jun. 3, 1919 and 1362850 of Dec. 21, 1920 to A. L. Clapp; 2024594 of Jan. 7, 1936 to H. W. Richter; **Can. Pat.** 173917 of Dec. 12, 1916 to C. N. Forrest, R. L. Fowler and M. S. Darrow; **Brit. Pats.** of 1801 (Jun. 3), 2507 to Thomas Winter; of 1864 (Sep. 16), 2271 to J. B. Wood.

27 [p. 622] **U. S. Pats.** 1577955 of Mar. 23, 1926 to A. L. Clapp; **Can. Pats.** 248678 of Apr. 14, 1925 to A. L. Clapp; 249352 of May 5, 1925 to A. L. Clapp.

28 [p. 622] "Cotton Linters for Paper Making," by H. A. Lindenberg, *Paper*, 27, 26 and 36 (1921); **U. S. Pat.** 1529299 of Mar. 10, 1925 to A. L. Clapp.

29 [p. 622] **U. S. Pats.** 1526632 of Mar. 3, 1925 to P. D. Richards; 1673967 of Jun. 19, 1928 and 1674948 of Jun. 26, 1928 to A. L. Clapp; **Can. Pats.** 248676, 248677 and 248678 of Apr. 14, 1925 to A. L. Clapp.

30 [p. 622] **U. S. Pat.** 130222 of Aug. 6, 1872 to J. L. Kendall.

31 [p. 622] **U. S. Pat.** 1511475 of Oct. 14, 1924 to K. B. Howell and C. R. Eckert.

32 [p. 622] **U. S. Pat.** 1649984 of Nov. 22, 1927 to A. W. Schorger.

33 [p. 622] "Füllstoffe für Bitumenmassen," by A. W. Rick, *Teer u. Bitumen*, 31, 390 (1935); **U. S. Pats.** 1256541 of Feb. 19, 1918 to William Fay; 1656828 of Jan. 5, 1927 to E. R. Powell; 1824430 of Sep. 22, 1931 to C. C. Hall; 1899056 of Feb. 28, 1933 to E. R. Powell; 1948395 of Jan. 16, 1928 to E. R. Powell; Reissue 19627 of Jun. 25, 1935 to E. R. Powell; **Brit. Pat.** of 1877 (Jun. 8), 2236 to A. M. Clark.

34 [p. 622] See Chapter 24; Reference 107.

35 [p. 622] **U. S. Pat.** 1463611 of Jul. 31, 1923 to A. L. Clapp.

36 [p. 622] **U. S. Pat.** 436733 of Sep. 16, 1890 to J. W. Easton.

37 [p. 622] **U. S. Pats.** 1305770 of Jun. 3, 1919 to A. L. Clapp; 1803409 and 1803410 of May 5, 1931 to M. O. Schur; **Can. Pat.** 319446 of Feb. 2, 1930 to Brown Co.

38 [p. 622] **U. S. Pats.** 81641 of Sep. 1, 1868 to H. W. Johns; 1698298 of Jan. 8, 1929 to A. L. Clapp; **Can. Pat.** 270963 of May 24, 1927 to A. L. Clapp; **Brit. Pat.** of 1881 (Sep. 26), 4687 to S. Pitt.

39 [p. 622] **U. S. Pat.** 300946 of Jun. 24, 1884 to Philip Carey.

40 [p. 622] "Standards for Products of the Asphalt Industry," by Julius Marcusson, *Mitt. k. Materialprüfungsamt*, 34, 40 (1916); also *J. Soc. Chem. Ind.*, 35, 1099 (1917).

41 [p. 623] The "breaking weight" represents the weight which must be applied to break a strip of given width.

42 [p. 623] **U. S. Pats.** 206436 of Jul. 30, 1878 to J. H. Greene; 296163 of Apr. 1, 1884 to Levi Haas and Dennis Howarth; 1278270 of Sep. 10, 1918 to H. L. Wilber; 1284997 of Nov. 19, 1918 to O. A. Bigler; 1348259 of Aug. 3, 1920 to H. L. Wilber; 1788121 of Jan. 6, 1931 to F. C. Overbury; **Can. Pat.** 190330 of May 13, 1919 to H. L. Wilber; **Brit. Pat.** 127536 of Jan. 31, 1919 to G. W. Johnson; **Ger. Design Pat.** 1213324 of Mar. 15, 1932 to A. W. Andernach.

43 [p. 623] **U. S. Pats.** 1317182 of Sep. 30, 1919 to A. S. Spiegel; 1493447 of May 6, 1924 to O. A. Heppes; **Can. Pat.** 210449 of Apr. 12, 1921 to Roofing Patents Co.

44 [p. 623] **U. S. Pat.** 1311862 of Jul. 29, 1919 to F. W. Adams.

45 [p. 623] **U. S. Pats.** 1331365 of Feb. 17, 1920, 1369186 and 1369187 of Feb. 22, 1921 to R. P. Perry; 1976948 of Oct. 16, 1934 to T. B. Lehon; 2031950 of Feb. 25, 1936 to N. P. Harshberger.

46 [p. 623] **U. S. Pats.** 1589513 of Jun. 22, 1926 to A. L. Clapp; 1643373 of Sep. 27, 1927 to A. L. Clapp; 1802494 of Apr. 28, 1921 to H. C. Avery; 1852006 of Apr. 5, 1932 to H. A. Emery; 1979819 of Nov. 6, 1934 to B. C. Beckman and W. T. Hofmann; 1995904 of Mar. 26, 1935 to J. C. Sherman; 2033488 of Mar. 10, 1936 to J. C. Sherman; 2048045 of Jul. 21, 1936 to F. W. Adams; **Can. Pats.** 278172 of Feb. 28, 1928 to Flintkote Co.; 343125 of Jul. 17, 1934

to Brown Co.; 345960 of Nov. 13, 1934 to Brown Co.; 350703 of Jun. 4, 1935 to Barrett Co.; 352604 of Aug. 27, 1935 to Brown Co.; 356762 of Mar. 24, 1936 to Brown Co.

47 [p. 623] **U. S. Pat.** 2024600 of Dec. 17, 1935 to G. A. Richter and M. O. Schur.

48 [p. 623] **U. S. Pat.** 1943257 of Jan. 9, 1934 to N. P. Harshberger.

49 [p. 623] **U. S. Pat.** 1352687 of Sep. 14, 1920 to R. P. Perry.

50 [p. 623] "Fireproofing Paper and Boards," by Joseph Rossman, *Paper Trade J.*, 91, 42 (1930).

51 [p. 623] **U. S. Pats.** 199656 of Jan. 29, 1878 to Oscar Mayo; 363905 of May 31, 1887 to J. G. Merrill; 560614 of May 19, 1896 to J. F. Geisler; 1612678 of Dec. 28, 1926 to F. S. Vivas; 1945714 of Feb. 6, 1934 to A. Winogradow; **Brit. Pats.** of 1888 (Dec. 21), 18708 to E. Edwards; of 1889 (Apr. 6), 7237 to A. Camille; **Ger. Pat.** 228706 of Jun. 22, 1909 to Sally Lissauer.

52 [p. 623] "The Effect of Certain Fireproofing Solutions on Cotton Fabric," by R. L. Sibley, *J. Ind. Eng. Chem.*, 13, 676 (1921); **U. S. Pats.** 60635 of Dec. 18, 1866 to Thos. Irving, John McNeil, G. W. Rich and C. J. Field; 1116349 of Nov. 3, 1914 to W. C. Zeller; 1165062 of Dec. 21, 1915 to A. C. Borzner; 1382618 of Jun. 28, 1921 to Guido Blenio; 1418730 of Jun. 6, 1922 to R. E. Ottman; 1612678 of Dec. 28, 1926 to F. S. Vivas; 1860097 of May 24, 1932 to W. B. Hoggatt.

53 [p. 623] **U. S. Pats.** 1778147 of Oct. 14, 1930 to E. S. Edwards; 1860134 of May 24, 1932 to D. M. Sutherland, Jr.

54 [p. 623] **U. S. Pat.** 101101 of Mar. 22, 1870 to J. R. Cole.

55 [p. 623] **U. S. Pats.** 52553 of Feb. 13, 1866 to A. L. Fleury; 225858 of Mar. 23, 1880 to C. H. O'Connor; 363905 of May 31, 1887 to J. G. Merrill; 671548 of Apr. 9, 1901 to Isaac Gordon; 724788 of Apr. 7, 1903 to Guido Blenio; 921002 of May 11, 1909 to I. L. Roberts; 970643 of Sep. 20, 1910 to I. L. Roberts; 1165062 of Dec. 21, 1915 to A. C. Borzner; 1273213 of Jul. 23, 1918 to C. F. Frohe; **Ger. Pats**, 328759 of May 10, 1919 to Dr. Plönnis & Co.; 443644 of Oct. 30, 1919 to Dr. Plönnis & Co.

56 [p. 623] **U. S. Pats.** 560614 of May 19, 1896 to J. F. Geisler; 1393355 of Oct. 11, 1921 to J. C. Emhardt; **Ger. Pat.** 196322 of Jan. 12, 1905 to Louis Schwabe.

57 [p. 623] **U. S. Pats.** 1199656 of Jan. 29, 1878 to Oscar Mayo; 724788 of Apr. 7, 1903 to Guido Blenio; 1116349 of Nov. 3, 1914 to W. C. Zeller; 1261736 of Apr. 2, 1918 to G. E. Ferguson; 1418730 of Jun. 6, 1922 to R. E. Ottman; 1577890 of Mar. 23, 1926 to C. D. Abraham; 1691726 of Nov. 13, 1928 to Charles Lichtenstadt; 1786270 of Dec. 23, 1930 to H. M. Spencer; 1793357 of Feb. 17, 1931 to C. H. Brown.

58 [p. 623] **U. S. Pat.** 1837150 of Dec. 15, 1931 to Camille Dreyfus and Herbert Platt.

59 [p. 623] **U. S. Pat.** 655845 of Aug. 14, 1900 to A. G. Winter.

60 [p. 623] **U. S. Pat.** 1534389 of Apr. 21, 1925 to A. C. Holzapfel.

61 [p. 623] **U. S. Pat.** 1338322 of Apr. 27, 1920 to T. B. Munroe.

62 [p. 623] "Fireproofing of Fiber Boards," by Mark Plunguian and E. C. Jahn, Proc. 5th Pacific Sci. Congr., 5, 3923 (1934); **U. S. Pat.** 1924181 of Aug. 29, 1933 to R. W. Cutler.

63 [p. 624] **U. S. Pats.** 318433 of May 19, 1885 to A. E. Newman; 1821317 of Sep. 1, 1931 to H. R. Offord and N. T. Mirov.

64 [p. 624] **Japan Pat.** 35965 of Mar. 11, 1920 to Y. Sakakibara.

65 [p. 624] **U. S. Pat.** 1804417 of May 12, 1931 to Otto Herting.

66 [p. 624] **U. S. Pat.** 675910 of Jun. 11, 1901 to Alwin Nieske.

67 [p. 624] **U. S. Pat.** 1335901 of Apr. 6, 1920 to T. B. Munroe.

68 [p. 624] **U. S. Pat.** 1280400 of Oct. 1, 1918 to A. L. Clapp.

69 [p. 624] **U. S. Pat.** 1839135 and 1839136 of Dec. 29, 1931 to F. S. Vivas.

70 [p. 624] **U. S. Pat.** 1995623 of Mar. 26, 1935 to G. A. Richter.

71 [p. 624] **Brit. Pat.** of 1889 (Jul. 11), 11149 to G. Rogers and J. Rogers.

72 [p. 624] "Preliminary Study of the Fireproofing of Fiber Boards," by Mark Plunguian and E. C. Jahn, Proc. 5th Pacific Sci. Congr., 5, 3923 (1934).

73 [p. 624] **U. S. Pats.** 73899 of Jan. 28, 1868 to Abiezer Jameson; 449636 of Mar. 31, 1891 to J. A. Smith; **Ger. Pats.** Design 1243956 of Aug. 25, 1932 to Papierfabrik Krampe, A.-G.; Design 1243978 of Nov. 22, 1932 to Papierfabrik Krampe, A.-G.

74 [p. 624] "Die Herstellung von Asbestpappe und Asbestpapier," by K. A. Weniger, A. Hartleben's Verlag, Vienna and Leipzig (1920).

75 [p. 624] **U. S. Pats.** 230946 of Aug. 10, 1880 to H. W. Johns; 248324 of Oct. 18, 1881 to H. W. Johns; **Brit. Pat.** of 1881 (Oct. 26), 4687 to S. Pitt.

76 [p. 624] **U. S. Pats.** 1689985 of Oct. 30, 1928 to H. R. Wardell; 1690079 of Oct. 30, 1928 to W. R. Seigle; 1796861 of Mar. 17, 1931 to G. D. Crabbs; **Can. Pat.** 233652 of Aug. 21, 1923 to W. R. Seigle.

77 [p. 624] **U. S. Pats.** 230946 of Aug. 10, 1880 to H. W. Johns; 1885113 of Nov. 1, 1932 to Thomas Jenkins; **Brit. Pat.** 184609 of Jun. 24, 1921 to N. Sulzberger.

78 [p. 624] "Chrysotile Asbestos in Canada," by J. G. Ross, Ottawa, Can. (1931).

79 [p. 625] **U. S. Pats.** 218389 of Aug. 12, 1879 to H. S. Lucas; 300946 of Jun. 24, 1884 to Philip Carey; 363905 of May 31, 1887 to J. G. Merrill; 1523634 of Jan. 20, 1925 to R. B. Crabbs; 1558495 of Oct. 27, 1925 to F. C. Overbury; 1577074 of Mar. 16, 1926 to R. P. Perry; 1687599 of Oct. 16, 1928 to C. A. Upson; **Can. Pat.** 249093 of Apr. 28, 1925 to Flintkote Co.

80 [p. 625] **U. S. Pat.** 1887726 of Nov. 15, 1932 to Louis Weber.

81 [p. 625] "Federal Spec. for Drill; Unbleached," Federal Standard Stock Catalogue, Section IV, (Part 5), CCC-D-651, Jun. 28, 1932; Bureau of Standards Circular C-367.

82 [p. 625] "Handbook of Industrial Fabrics," by G. B. Haven, Wellington Sears Co., New York (1935); "Standard Specifications for Tolerances for Numbered Cotton Duck" (D 230–27), A.S.T.M. Standards 1936, II, 1293; "Standard Methods of Testing and Tolerances for Certain Light and Medium Weight Cotton Fabrics" (D 274–36), A.S.T.M. Standards 1936, II, 1365; "Standard Methods of Testing and Tolerances for Hose Ducks and Belt Ducks" (D 181–36), A.S.T.M. Standards 1936, II, 1362; "Tentative Definitions of Terms Relating to Textile Materials" (D 123–36T), A.S.T.M. Tentative Standards 1936, 1253; "Federal Specification for Textiles; Test Materials," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-T-191, May 2, 1933; "Federal Specification for Duck; Cotton (Canvas), Light-weight, Unbleached (Army-Duck)," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-D-751, Feb. 7, 1933; "Federal Specification for Duck; Cotton, Plied-Filling-Yarns and Single-Yarns (Flat-Duck)," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-D-761, Feb. 7, 1933; "Federal Specification for Duck; Cotton, Plied-Yarns (Army, Numbered, and Tent-Duck)," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-D-771, May 31, 1932; "Federal Specification for Duck; Cotton, Numbered," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-D-831, Jun. 16, 1934.

83 [p. 625] "Federal Spec. for Burlap; Jute," Federal Standard Stock Catalogue, Section IV, (Part 5), CCC-B-811, Feb. 15, 1933; Bureau of Standards Circular C-352.

84 [p. 625] **U. S. Pat.** 1352931 of Sep. 14, 1930 to Oscar Altpeter; **Brit. Pat.** of 1897 (Jan. 12), 809 to Eduard Kauert.

85 [p. 626] "Bitumen for Impregnating and Saturating," by Fritz Hoyer, *Farben-Chem.*, **6**, 331 (1935).

86 [p. 626] **Can. Pats.** 247333 of Mar. 3, 1925 to Flintkote Co.; 282444 of Aug. 14, 1928 to James Reid.

87 [p. 626] **U. S. Pat.** 1916584 of Jul. 4, 1933 to Wilhelm Pungs; **Ger. Pat.** 391148 of Jan. 26, 1923 to Warnecke & Böhm, A.-G.

88 [p. 626] **U. S. Pats.** 248074 of Oct. 11, 1881 to C. M. Warren; 356411 of Jan. 18, 1887 to Terrence McSweeney.

, 89 [p. 626] **Ger. Pats.** 24712 of Apr. 6, 1883 to Carl Lortzing; 30318 of Feb. 12, 1884 to Carl Lortzing.

90 [p. 626] **U. S. Pats.** 1433137 of Oct. 24, 1922 to Hermann Kambach; **Brit. Pats.** of 1883 (Dec. 22), 5845 to J. L. Clark; of 1907 (Jun. 17), 13943 to W. A. Phillips and Frederick Hutchins; of 1908 (Mar. 9), 5279 to E. S. Ali-Cohen; 167757 and 167759 of Aug. 6, 1921 to Hermann Kambach; **Ger. Pats.** 344992 of Jan. 30, 1919 to Hermann Kambach; 350820 of Aug. 15, 1919 to Hermann Kambach; 355373 of Feb. 6, 1919 to Hermann Kambach; **French Pat.** 533328 of Mar. 17, 1921 to Hermann Kambach.

91 [p. 626] **U. S. Pats.** 283200 of Aug. 14, 1883 to H. R. Brissett and John Howe; 1163593 of Dec. 7, 1915 to C. N. Forrest; **Can. Pat.** 171345 of Aug. 15, 1916 to C. N. Forrest; **Brit. Pat.** of 1915 (Nov. 12), 15979 to Barber Asphalt Paving Co.; **Ger. Pats.** 154220 of Jul. 9, 1902 to Franz Horn; 294046 of Dec. 3, 1915 to Barber Asphalt Paving Co.; **French Pat.** 480271 of Jul. 12, 1916 to Barber Asphalt Paving Co.

92 [p. 626] **U. S. Pat.** 283044 of Aug. 14, 1883 to D. B. Turner.

93 [p. 626] **Ger. Pats.** 552751 of Mar. 9, 1928 to Kaspar Winkler; 562808 of Mar. 9, 1928 to Kaspar Winkler.

94 [p. 626] **U. S. Pat.** 376289 of Jan. 10, 1888 to Heinrich Busse.

95 [p. 626] **Brit. Pat.** 362034 of Sep. 15, 1930 to I. G. Farbenindustrie, A.-G.

96 [p. 626] **Brit. Pat.** 178558 of Jan. 18, 1921 to Frederick Lamplough.

97 [p. 626] **U. S. Pats.** 1969904 of Aug. 14, 1934 to S. S. Sadtler; 2005113 of Jun. 18, 1935 to Orvall Smiley.

98 [p. 626] "Normen für Teer Imprägniermasse und Pech zur Dachpappenfabrikation," *Teer*, 3, 25 (1922); *Teer*, 21, 115 and 127 (1923); "Teer und Pech," by Heinrich Mallison, *Teer*, 22, 13 (1924).

99 [p. 626] **U. S. Pats.** 75128 of Mar. 3, 1868 to Francis Curtis; 101406 of Mar. 29, 1870 to C. H. Smith.

100 [p. 626] **Ger. Pats.** Design 339008 of Apr. 22, 1908 to Fritz Grimm; 284886 of Oct. 23, 1913 to Schatz & Hübner.

101 [p. 626] **U. S. Pat.** 1796861 of Mar. 17, 1931 to G. D. Crabbs.

102 [p. 626] **Brit. Pat.** of 1900 (Dec. 24), 23603 to P. C. Ralli, H. Mayer and L. Toch.

103 [p. 626] **U. S. Pat.** 371681 of Oct. 18, 1887 to John Grant; **Ger. Pat.** 597708 of May 29, 1931 to Bruno Peckie.

104 [p. 626] **U. S. Pat.** 621807 of Mar. 28, 1899 to B. Ford.

105 [p. 626] **U. S. Pat.** 1179357 of Apr. 11, 1916 to Kurt Haring.

106 [p. 626] **U. S. Pats.** 254461 of Mar. 7, 1882 to J. D. Cheever; 319079 of Jun. 2, 1885 to J. W. Ellis; 362076 of May 3, 1887 to S. H. Gilson; 391927 of Oct. 30, 1888 to J. A. Titzel; 533440 of Feb. 5, 1895 to C. H. Dalrymple; 1391934 of Sep. 27, 1921 to N. C. Bissel; **Brit. Pat.** 380693 of Jan. 25, 1932 to Adele Thielmann; **Ger. Pats.** 81565 of Jul. 12, 1894 to Rudolph Wiggert; Appl. St-42.30 of Nov. 25, 1930 to Hermann Stellwag.

107 [p. 626] **U. S. Pats.** 387357 and 387358 of Aug. 7, 1888 to M. S. Higbie and A. W. Dougherty.

108 [p. 627] **U. S. Pats.** 205135 of Jun. 18, 1878 to W. H. Rankin; 1720780 of Jul. 16, 1929 to C. S. Cooley; 1992240 of Feb. 26, 1935 to S. S. Sadtler.

109 [p. 627] **U. S. Pats.** 190953 of May 22, 1877 to Charles Brown; 398337 of Feb. 19, 1889 to W. B. Lupton.

110 [p. 627] **Ger. Pat.** 121436 of May 6, 1899 to A. W. Andernach.

111 [p. 627] **U. S. Pat.** 273233 of Feb. 27, 1883 to W. H. Horner and Francis Hyde.

112 [p. 627] **Ger. Pat.** 122893 of May 5, 1899 to A. W. Andernach.

113 [p. 627] **Brit. Pat.** 156636 of Nov. 3, 1921 to Société Veuve Etienne Baron et Cie.; **Ger. Pat.** 100700 of Apr. 27, 1897 to Carl Baswitz.

114 [p. 627] **U. S. Pat.** 1789284, 1789285 and 1789286 of Jan. 13, 1931 to A. C. Fischer.

115 [p. 627] **Brit. Pat.** of 1858 (Mar. 26), 639 to P. H. G. Bérard.

116 [p. 627] **Ger. Pats.** 94172 of May 5, 1895 to Carl Baswitz; 100700 of Apr. 27, 1897 to Carl Baswitz.

117 [p. 627] **U. S. Pat.** 204829 of Jun. 11, 1878 to W. P. Jenney; **Ger. Pat.** 198470 of Jan. 31, 1907 to Chem. Fabrik Flörsheim, Dr. H. Noerdlinger A.-G.; **Aust. Pat.** 22226 of Dec. 3, 1904 to Posnansky & Strelitz.

118 [p. 627] **U. S. Pats.** 1417838 of May 30, 1922 to Lester Kirschbraun; 1528436 of Mar. 3, 1925 to Lester Kirschbraun.

119 [p. 627] **U. S. Pat.** 1549991 of Aug. 18, 1925 to Lester Kirschbraun.

120 [p. 627] **U. S. Pats.** 253656 of Feb. 14, 1882 to S. M. Allen; 319806 of Jun. 9, 1885 to J. F. Fogg; 398337 of Feb. 19, 1889 to W. B. Lupton; 674219 of May 14, 1901 to J. A. Scharwath; 1310624 of Jul. 22, 1919 to H. F. Kenworthy.

121 [p. 627] **U. S. Pat.** 1562382 of Nov. 17, 1925 to C. M. Taylor.

122 [p. 628] **U. S. Pat.** 1811613 of Jun. 23, 1931 to A. L. Clapp.

123 [p. 628] "Die Herstellung feuersicherer Dachpappe," by Dr. Sedlaczek, *Teer*, 24, 279 (1926).

124 [p. 628] **U. S. Pats.** 1812732 of Jun. 30, 1931 to J. H. Young; 1975072 of Oct. 2, 1934 to C. F. Booth; 1994810 of Mar. 19, 1935 to Marc Darrin; 2025929 of Dec. 31, 1935 to J. H. Young; **Can. Pats.** 319521 of Feb. 2, 1932 to H. H. Robertson Co.; 327878 of Nov. 22, 1932 to H. H. Robertson Co.; 341792 of May 22, 1934 to Canadian Industries, Ltd.; **Brit. Pats.** 371683 of Jun. 30, 1931 to J. H. Young; 401401 of Nov. 16, 1933 to J. H. Young; **Ger. Pat.** 567261 of Jun. 4, 1931 to I. G. Farbenindustrie, A.-G.

125 [p. 628] **U. S. Pats.** 914222, 914223, 914224 of Mar. 2, 1909 to J. W. Aylsworth; 914251 of Mar. 2, 1909 to Carleton Ellis and K. P. McElroy; 914300 of Mar. 2, 1909 to K. P. McElroy and Carleton Ellis; 1028926 and 1029254 of Jun. 11, 1912 to J. W. Aylsworth; 1077113 of Oct. 28, 1913 to J. W. Aylsworth; 1083354 of Jan. 6, 1914 to T. A. Edison; 1084520 of Jan. 13, 1914 to K. L. V. Zimmer; 1087422 of Feb. 17, 1914 to J. W. Aylsworth; 1090439 of Mar. 17, 1914 to J. W. Aylsworth; 1155130 of Sep. 28, 1915 and 1162453 of Nov. 30, 1915 to S. R. Church; 1183423 of May 16, 1916 to J. W. Aylsworth; 1196505 of Aug. 29, 1916 to J. W. Aylsworth; 1246810 of Nov. 13, 1917 to Carleton Ellis; 1248638 of Dec. 4, 1917 to J. W. Aylsworth; 1398991 of Dec. 6, 1921 to J. H. Young; 1928105 of Sep. 28, 1933 to Ludwig Kern; 2030653 of Feb. 11, 1936 to R. G. Quinn; **Can. Pats.** 220779 of Jul. 11, 1922 to H. H. Robertson Co.; 303839 of Sep. 9, 1930 to Halowax Corp.; 349742 of Apr. 23, 1935 to Canadian Industries, Ltd.; **Brit. Pat.** 186861 of Dec. 6, 1921 to J. H. Young; **Ger. Pats.** 537595 of Apr. 13, 1926 to I. G. Farbenindustrie A.-G.; Appl. J-42028 of Jul. 8, 1931 to I. G. Farbenindustrie A.-G.; Appl. J-42666 of Sep. 24, 1931 to I. G . Farbenindustrie A.-G.; Appl. J-45469 of Oct. 8, 1932 to I. G. Farbenindustrie A.-G.; Appl. J-45470 of Oct. 8, 1932 to I. G. Fabenindustrie A.-G.; 595461 of Jan. 13, 1929 to I. G. Farbenindustrie A.-G.; 610099 of Mar. 2, 1935 to Verein für chem. und metallurgische Produktion; **Fr. Pat.** 544242 of Dec. 6, 1921 to H. H. Robertson Co.

126 [p. 628] **U. S. Pat.** 1194558 of Aug. 15, 1916 to J. V. Skoglund.

127 [p. 628] **U. S. Pats.** 1388826, 1328827, 1388828 and 1388829 of Aug. 23, 1921 to Arthur Arent; **Can. Pat.** 229126 of Feb. 27, 1923 to Arthur Arent Laboratories, Inc.; **Brit. Pats.** 132813 of Sep. 20, 1919 and 146099 of Jun. 26, 1919 to Arthur Arent.

128 [p. 628] **U. S. Pat.** 1167195 of Jan. 4, 1916 to Oskar Matter; **Brit. Pat.** of 1913 (Jan. 20), 1155 to Alfred Maschke; **Ger. Pat.** 267407 of Jan. 21, 1913 to Alfred Maschke.

129 [p. 628] **U. S. Pats** 1641637 of Sep. 6, 1927 to L. C. Lutyens and R. O. Child; **Brit. Pats.** 238586 of Apr. 11, 1924 to L. C. Lutyens; 286950 of Apr. 14, 1927 to R. O. Child.

130 [p. 628] **U. S. Pat.** 1612677 and 1612678 of Dec. 28, 1926 to F. S. Vivas.

131 [p. 628] **Can. Pat.** 8124 of Nov. 17, 1877 to J. B. Slichter; **Ger. Pat.** 228706 of Nov. 14, 1910 to Sally Lissauer.

132 [p. 628] **U. S. Pat.** 1534389 of Apr. 21, 1925 to A. C. Holzapfel.

133 [p. 628] **U. S. Pat.** 433215 of Jul. 29, 1890 to Israel Rabinowicz.

134 [p. 628] **Can. Pats.** 325758 and 325759 of Sep. 6, 1932 to International Paper Co.

135 [p. 628] **U. S. Pats.** 1715080 of May 28, 1929 to O. A. Frederickson.

136 [p. 628] **U. S. Pats.** 307184 of Oct. 28, 1884 to A. Derrom; 327477 of Sep. 29, 1885 to H. C. Spaulding.

137 [p. 628] **Brit. Pat.** of 1888 (Apr. 14), 5577 to W. P. Thompson.

138 [p. 628] **U. S. Pats.** 267045 of Nov. 7, 1882 to R. S. Waring and J. B. Hyde; 267046 of Nov. 7, 1882 to R. S. Waring; Reissue 10350 of Jul. 3, 1883 to R. S. Waring and J. B. Hyde; Reissue 10351 of Jul. 3, 1883 to R. S. Waring; 284098 of Aug. 28, 1883 to R. S. Waring. **Austrian Pat.** 5448 of Oct. 10, 1901 to Hans Blechschmidt.

139 [p. 628] **U. S. Pats.** 17851 of Jul. 21, 1857 to J. B. Wands; 19802 of Mar. 30, 1858 to Joseph Thompson; 128599 of Jul. 2, 1872 to W. B. Davies; 150636 of May 5, 1874 to J. A. Turner; 395394 of Jan. 1, 1889 to John Hunter; 423042 of Mar. 11, 1890 to A. N. Ford; 447412 of Mar. 3, 1891 to J. S. Palmer; 1016801 of Feb. 6, 1912 to L. S. Van Westrum; 1358371 of Nov. 9, 1920 to A. C. Holzapfel; 1855743 of Apr. 26, 1932 to S. H. Hartman;

Brit. Pats. of 1863 (Jun. 25), 1603 to William Kirrage; of 1867 (Sep. 21), 2656 to G. E. Marchisio; of 1868 (Apr. 23), 1336 to Joseph Rogers; of 1868 (Jun. 13), 1932 to Charles Humfrey; of 1872 (Aug. 29), 2572 to W. R. Lake; of 1873 (Mar. 19), 1007 to George Newton; of 1873 (Sep. 26), 3147 to J. A. Turner; of 1874 (Feb. 4), 447 and 449 to John Macintosh; of 1876 (Mar. 27), 1309 to Caleb Tayler; of 1877 (Jun. 28), 2498 to J. W. Jeune and J. Barnard; of 1886 (May 14), 6518 to J. Trotter; of 1887 (Dec. 19), 17442 to A. N. Ford; of 1888 (Mar. 5), 3354 to Thomas Thomson; of 1888 (Jul. 5), 9761 to J. S. Palmer; of 1889 (May 27), 8795 to A. N. Ford; of 1893 (Sep. 9), 17003 to Emille Pierret; of 1897 (Apr. 1), 8343 to J. D. Blackwell; of 1907 (Mar. 27), 7373 to C. A. Peck; **Aust. Pat.** 56296 of Nov. 11, 1912 to Wenzel Massag.

140 [p. 628] **U. S. Pat.** 633255 of Sep. 19, 1899 to E. J. Yetter.

141 [p. 628] **U. S. Pats.** 617706 of Jan. 17, 1899 to C. E. Anthony; 620082 of Feb. 21, 1899 to C. E. Anthony.

142 [p. 628] **U. S. Pat.** 1510472 of Oct. 7, 1924 to F. W. Farrell; **Brit. Pat.** of 1913 (Jun. 25), 14664 to H. Merrylees.

143 [p. 628] **U. S. Pats.** 1330443 of Feb. 10, 1920 to G. C. Glynn; 1448155 of Mar. 13, 1923 to Karl Schutté; 1464717 of Aug. 14, 1923 to Lester Kirschbraun; 1489254 of Apr. 8, 1924 to Lester Kirschbraun; 1494380 of May 20, 1924 to Karl Schutté; 1723361 of Aug. 6, 1929 to H. L. Levin; 1724137 of Aug. 13, 1929 to Lester Kirschbraun and C. R. MacDonald; 1865634 of Jul. 5, 1932 to Lester Kirschbraun; **Can. Pats.** 302330 of Jul. 22, 1930 to Flintkote Co.; 307695 of Jan. 13, 1931 to Flintkote Co.; 332018 of Apr. 25, 1933 to Flintkote Co.; **Brit. Pats.** 319212 and 319213 of Sep. 16, 1929 to N. V. de Bataafsche Petroleum Maatschappij; **Ger. Pat.** Appl. E-18313 of Aug. 8, 1912 to Ludwig Esselborn (rejected); **Russian Pat.** 33267 of Nov. 30, 1933 to A. I. Nazarov.

144 [p. 628] **U. S. Pat.** 1450712 of Apr. 3, 1923 to W. H. Cady; **Can. Pat.** 249586 of May 12, 1925 to Flintkote Co.

145 [p. 628] **U. S. Pats.** 1450712 of Apr. 3, 1923 to W. H. Cady; 1623189 of Apr. 5, 1927 to Lester Kirschbraun and W. H. Cady.

146 [p. 629] **Ger. Pats.** Design 1255161 of Feb. 23, 1933 to Binné & Sohn; Design 1267722 and 1267723 of Jun. 9, 1933 to Vedag, Vereinigte Dachppappen-Fabriken, A.-G.

147 [p. 629] **U. S. Pat.** 1510591 of Oct. 7, 1924 to F. W. Farrell; **Brit. Pat.** 382419 of Oct. 23, 1931 to H. Hands; **Ger. Pat.** Appl. W-78737 of Mar. 8, 1928 to Kaspar Winkler; 615175 of May 19, 1931 to Deutsche Röhrenwerke A.-G.

148 [p. 629] **U. S. Pat.** 678922 of Jul. 23, 1901 to C. W. Walker; **Can. Pat.** 74994 of Mar. 4, 1902 to C. H. Walker.

149 [p. 629] **U. S. Pat.** 1984922 of Dec. 18, 1934 to W. C. Fischer; **Ger. Pats.** 591539 of Feb. 19, 1931 to Adele Thielmann; 613315 of Feb. 19, 1931 to Adele Thielmann; 615175 of Jun. 28, 1935 to Deutsche Röhrenwerke A.-G.; **French Pat.** 783041 of Jul. 6, 1935 to Internationale Vereeniging Voor de Rubber-En Andere Cultures in Nederlandsch-Indië.

150 [p. 629] **U. S. Pats.** 1500537 of Jul. 8, 1924 to N. A. Thompson; 1502598 of Jul. 22, 1924 to N. A. Thompson; **Brit. Pat.** 377862 of Nov. 6, 1931 to F. T. Lakin, J. Leach and H. Cartlidge.

151 [p. 629] **Brit. Pat.** 399147 of Sep. 15, 1933 to John Lightfoot.

152 [p. 629] **Ger. Pat.** Appl. A-59309 of Oct. 11, 1929 to Amber Size & Chemical Co., Ltd.

153 [p. 629] **Can. Pat.** 328432 of Dec. 13, 1932 to Bakelite, G.m.b.H.

154 [p. 630] "Der Paraffingehalt des Bitumens," by Paul Riehm, *Teer u. Bitumen*, **29**, 529 (1931); "The Properties of Bitumen Poor and Rich in Paraffin," by J. Manheimer, *Petroleum Z.*, **28**, 1 (1932); "Ueber die Einwirkung des Paraffin- und Schwefelgehalts im Erdölpech auf die Duktilität inbesondere bei russischen Bitumina," by I. B. Longinus, *Teer u. Bitumen*, **30**, 357 (1932); "Influence of Paraffin Wax on the Properties of Asphaltic Bitumen," by Paul Riehm, Proc. World Petroleum Congress, **2**, 552 (1933); "Influence of Paraffin Wax on Bitumen," by J. Manheimer, Proc.World Petroleum Congress **2**, 553 (1933).

155 [p. 631] **U. S. Pats.** 107967 of Oct. 4, 1870 to George Shove; 791312 of May 30, 1905

to C. S. Bird and G. D. Moore; 1187259 of Jun. 13, 1916 to B. G. Casler; 1273673 of Jul. 23, 1918 to Jean Rosen; 1801245 of Apr. 14, 1931 to E. L. Chamberlain.

156 [p. 631] See Chapter 24, Reference 2.

157 [p. 631] "Farbige Bitumenerzeugnisse," by C. R. Platzmann, *Asphalt u. Teer Strassenbautechnik*, 32, 809 and 884 (1932); "Asphalt Bitumens for the Protection of Buildings," by C. R. Platzmann, *Bautenschutz*, 6, 19 (1935); **U. S. Pats.** 35720 of Jun. 24, 1862 to Charles Wanzer; 204829 of Jun. 11, 1878 to W. P. Jenney; 447412 of Mar. 3, 1891 to J. S. Palmer; 460249 of Sep. 29, 1891 to R. F. Flynn; 775635 and 775636 of Nov. 22, 1904 to L. C. Rugen and Herbert Abraham; 1131317 of Mar. 9, 1915 to C. S. Bird; **Can. Pats.** 30612 of Jan. 23, 1889 to C. T. Snedeker; 93027 of May 9, 1905 to Standard Paint Co.; 93160 of May 16, 1905 to Standard Paint Co.; 242208 and 242209 of Aug. 19, 1924 to Harold Goodwin; **Brit. Pats.** of 1904 (Feb. 6), 28488 to L. C. Rugen and Herbert Abraham; of 1904 (May 27), 28489 to L. C. Rugen and Herbert Abraham; of 1913 (Jun. 18), 14063 to Georg Halle; 212106 of Mar. 3, 1923 to Harold Goodwin and H. Smith; 212188 of Aug. 21, 1923 to Harold Goodwin and H. Smith; 218750 of Apr. 13, 1923 to A. G. Saunders; **Ger. Pats.** 199617 of Jan. 31, 1905 to Standard Paint Co.; 277643 of Jun. 6, 1913 to Friedrich Binder; 357611 of Aug. 26, 1918 to A. Blumenthal & Co.; 439519 of Jun. 26, 1921 to Ernst Stern; 640931 of Feb. 8, 1933 to Gesellschaft für Teerstrassenbau m.b.H.; Appl. D-58165 of Apr. 16, 1929 to Deutsche Asphalt A.-G.; **Fr. Pat.** 459344 of Jun. 17, 1913 to Société Posnansky & Strelitz; **Austrian Pat.** 60750 of Aug. 25, 1913 to Posnansky & Strelitz; **Hungarian Pat.** 115340 of Nov. 2, 1936 to Neuchatel Asphalt Co., Ltd.

158 [p. 631] "Ueber farbige Bitumenfabrikate," by C. R. Platzmann, *Teer und Bitumen*, 31, 369 (1933); "Colored Coatings for Roofs," by C. R. Platzmann, *Farbe u. Lack*, 233 and 243 (1935); "Asphalt Bitumens for the Protection of Buildings," by C. R. Platzmann, *Bautenschutz*, 6, No. 2, 19 (1935); *Bldg. Sci. Abstracts*, 8, 123 (1935).

159 [p. 631] **Can. Pat.** 330431 of Feb. 28, 1933 to J. D. Giles.

160 [p. 631] **U. S. Pat.** 103554 of May 31, 1870 to O. E. Bowen.

161 [p. 631] **U. S. Pat.** 1040706 of Oct. 8, 1912 to L. F. Lindley.

162 [p. 632] **Ger. Pat.** 447470 of Feb. 27, 1925 to Haba, G.m.b.H.

163 [p. 632] **U. S. Pat.** 205135 of Jun. 18, 1878 to W. H. Rankin.

164 [p. 632] **Ger. Pat.** 122893 of May 5, 1899 to A. W. Andernach.

165 [p. 632] **U. S. Pat.** 1788250 of Jan. 6, 1931 to O. O. Rieser.

166 [p. 632] **U. S. Pat.** 1164965 of Dec. 21, 1915 to H. S. Smalley.

167 [p. 632] "Streumittel für Dachpappen," by A. W. Rick, *Teer u. Bitumen*, 34, 97 (1936).

168 [p. 632] **Ger. Pats.** 92308 of Apr. 12, 1905 to A. W. Andernach; 298129 of Sep. 20, 1916 to Rudolf Plönnis & Co.; 328759 of May 10, 1919 to Rudolf Plönnis & Co.; 405299 of Oct. 30, 1924 to K. C. Menzel and Paul Meyersberg.

169 [p. 632] **Brit. Pat.** of 1887 (Sep. 22), 12684 to J. C. Lyman.

170 [p. 632] **U. S. Pat.** 686191 of Nov. 5, 1901 to W. H. Bache; **Ger. Pat.** 183552 of Nov. 18, 1904 to Hans Christen; **Swedish Pat.** 21509 of Nov. 19, 1904 to Hans Christen.

171 [p. 632] **U. S. Pat.** 775636 of Nov. 22, 1904 to L. C. Rugen and Herbert Abraham.

172 [p. 632] "Aluminized Roofing Paper," by Ernst Täuber, *Teer u. Bitumen*, 31, 387 (1933); **U. S. Pats.** 342315 of May 25, 1886 to Frederick Beck; 1574615 of Feb. 23, 1926 to C. S. Fleming; 1631604 of Jun. 7, 1927 to Thomas Robinson; **Can. Pats.** 261795 of Jun. 15, 1926 to Paraffine Cos., Inc.; 266310 of Nov. 30, 1926 to Flintkote Co.; **Brit. Pats.** 183143 of Jul. 17, 1922 to Katharina Wickel; 316241 of Apr. 26, 1928 to W. Briggs & Sons, Ltd. and A. S. Briggs; 414242 of Oct. 6, 1933 to F. Chabert; **Ger. Pats,** 290366 of Jan. 20, 1915 to Hans Christen; 496163 of Oct. 25, 1928 to Ernst Täuber; Appl. I-27754 of Mar. 24, 1926 to I. G. Farbenindustrie A.-G. (withdrawn); Appl. T-38898 of May 21, 1931 to Ernst Täuber; **Austrian Pat.** 31400 of Mar. 10, 1910 to Emil Kuznitzky; **Swedish Pat.** 74719 of Jul. 12, 1932 to K. J. T. Ohlsson.

173 [p. 632] **U. S. Pat.** 19627 of Mar. 16, 1858 to W. T. de Golver; **Austrian Pat.** 47413 of Apr. 10, 1911 to Max Kurz.

4 [p. 632] **Swedish Pat.** 71883 of May 26, 1931 to K. J. T. Ohlsson.

175 [p. 632] "Zonolite: Utilizing a Useless Mineral," by E. N. Alley, *Eng. and Mining J.*, **120**, 819 (1915); "Calcined Vermiculite as a Plaster Base," by C. S. Crouse, *Eng. Mining J.*, **128**, 923 (1929); "Vermiculite," by A. V. Petar, Bureau of Mines Information Circular No. 6720, Washington, D. C. (1933); "Vermiculite: Production and Marketing by the Zonolite Company," by W. S. Steele, *Trans. Am. Inst. Mining Met. Engrs.*, **109**, 418, (1934); "The Structure and Properties of Vermiculite," by V. P. Kazantsev, Mém. soc. russe minéral, **63**, 464 (1934); **U. S. Pats.** 67233 of Jul. 30, 1867 to J. R. Van Horn and J. B. Roberts; 1430085 of Sep. 26, 1922 to James Keeth; 1509406 and 1509478 of Sep. 23, 1924 to C. E. Kraus; 1693051 of Nov. 27, 1928 to J. A. Babor and W. L. Estabrooke; 1715977 of Jun. 4, 1929 to C. J. Bates, Jr. and C. P. Bartels; 1743551 of Jan. 14, 1930 to James Keeth; 1743744 of Jan. 14, 1930 to H. F. Winkelmann; 1778008 of Oct. 14, 1930 to J. H. Delaney; 1812306 of Jun. 30, 1931 to A. J. Russ; 1830253 of Nov. 3, 1931 to Paul Bechtner; 1839887 of Jan. 5, 1932 to G. A. New; 1892138 of Dec. 27, 1932 to E. R. Darling; 1898774 and 1898830 of Feb. 21, 1933 to R. G. Guthrie and O. J. Wilbor; 1905541 of Apr. 25, 1933 to J. A. Wiener and J. B. Harmon; 1919037 of Jul. 18, 1933 to W. S. Quigley; 1922448 of Aug. 15, 1933 to C. S. Miner; 1927102 of Sep. 19, 1933 to Glenn Sucetti and R. M. Kohr; 1950829 of Mar. 13, 1934 to G. M. Thomson; 1963275 of Jun. 19, 1934 to O. A. Labus; 1972390 of Sep. 4, 1934 to C. S. Miner; 1977874 of Oct. 23, 1934 to P. S. Denning; 1980889 of Nov. 13, 1934 to G. M. Thomson; 1989830 of Feb. 5, 1935 to J. T. Stroehlke; 1991393 of Feb. 19, 1935 to W. W. Joyce; 2009146 of Jul. 23, 1935 to G. A. New; 2017904 of Oct. 22, 1935 to James Keeth; 2018911 of Oct. 29, 1935 to P. B. Brill and G. H. Ellis; 2029524 of Feb. 4, 1936 to P. S. Denning; 2030239 of Feb. 11, 1936 to W. B. Byers; Reissue 19919 of Apr. 7, 1936 to Paul Bechtner; 2036913 of Apr. 7, 1936 to G. B. Brown and H. E. Holcomb; 2041910 and 2041911 of May 26, 1936 to Richard Ericson; 2043249 of Jun. 9, 1936 to O. L. Jones; 2047187 of Jul. 14, 1936 to H. L. Becher; 2058959 of Oct. 27, 1936 to P. S. Denning; 2063102 of Dec. 8, 1936 to O. L. Jones; 2076898 of Apr. 13, 1937 to O. A. Labus; 2077094 of Apr. 13, 1937 to William Byers; **Can. Pats.** 318020 of Dec. 15, 1931 to B. D. White; 337976 of Dec. 19, 1933 to American Colloid Co.; 347049 of Dec. 25, 1934 to National Vermiculite Products Corp.; 350811 of Jun. 11, 1935 to O. A. Labus; 353542 of Oct. 15, 1935 to G. M. Thomson; 356098 of Feb. 25, 1936 to O. A. Labus; 365298 of Apr. 13, 1937 to Glenn Sucetti; **Russian Pat.** 26392 of Apr. 11, 1929 to B. F. Puilaev.

176 [p. 632] **U. S. Pat.** 117196 of Jul. 18, 1871 to S. G. Morrison; **Ger. Pat.** Design 278651 of Apr. 2, 1906 to C. F. Weber Dachpappen und Teerproduktenfabrik.

177 [p. 632] **U. S. Pat.** 925263 of Jun. 15, 1909 to John Ayrault.

178 [p. 632] **U. S. Pats.** 56625 of Jul. 24, 1866 to John Snare; 109486 of Nov. 22, 1870 to Frederick Beck; 334850 of Jan. 26, 1886 to R. A. Denison; 925263 of Jun. 15, 1909 to John Ayrault; 1483711 of Feb. 12, 1924 to Maurice Blumenthal; **Can. Pat.** 51100 of Jan. 22, 1896 to Daniel Allen; **Ger. Pat.** Design 278651 of Apr. 2, 1906 to C. F. Weber.

179 [p. 632] **U. S. Pat.** 490668 of Jan. 31, 1893 to G. S. Lee; **Brit. Pats.** of 1868 (Oct. 24), 3249 to R. Ferguson and G. Lord; of 1877 (Mar. 16), 1060 to R. Taylor; of 1885 (Oct. 31), 13140 to J. E. A. Pierret; **Ger. Pats.** 207814 of Nov. 24, 1907 to Hermann Paul and Emil Ziaja; 239242 of Jun. 20, 1909 to Hermann Paul; 322986 of Aug. 8, 1916 to W. Kührmeyer-Franke & Klauer, G.m.b.H.; 305489 of Jul. 18, 1917 to Maschinenfabrik Calbe, A.S., G.m.-b.H.

180 [p. 632] **U. S. Pat.** 490668 of Jan. 31, 1893 to G. S. Lee.

181 [p. 632] **Ger. Pat.** Desig : 386979 of May 4, 1908 to Paul Riehm.

182 [p. 633] **U. S. Pats.** 310192 of Jan. 6, 1895 to J. T. Edson; 1007146 of Oct. 31, 1911 to E. J. Schroder; 1337822 of Apr. 20, 1920 to A. B. Cooke; **Brit. Pat.** of 1877 (Mar. 16), 1060 to R. Taylor; **Ger. Pat.** Appl. G-81049 of Nov. 4, 1931 to Karl Griesel.

183 [p. 633] **U. S. Pat.** 1075745 of Oct. 14, 1913 to S. G. Wright.

184 [p. 633] **U. S. Pat.** 1436914 of Nov. 28, 1922 to W. R. Seigle; **Can. Pat.** 242405 of Aug. 26, 1924 to W. R. Seigle.

185 [p. 633] "Paper Tiles," by Anon., *Scientific American*, **53** (New Series), No. 18, 272 (1885).

186 [p. 633] "Ceramic Roofing Granules," by E. H. Berger, *Ceramic Age*, **25**, 49 (1935).

187 [p. 633] **U. S. Pat.** 1022764 of Apr. 29, 1912 to S. G. Wright.

188 [p. 633] **U. S. Pats.** 311388 of Jan. 27, 1885 to R. H. Thompson; 334850 of Jan. 26, 1886 to R. A. Denison.

189 [p. 633] **U. S. Pats.** 1022764 of Apr. 9, 1912 to S. G. Wright; 1190505 of Jul. 11, 1916 to H. E. Boardman; 2037802 of Apr. 21, 1935 to E. A. Leonard; **Can. Pat.** 143027 of Sep. 24, 1912 to S. G. Wright; **Brit. Pat.** of 1912 (Apr. 1), 7872 to A. J. Boult.

190 [p. 633] **U. S. Pat.** 453979 of Jun. 9, 1891 to G. S. Lee.

191 [p. 633] **U. S. Pat.** 1430365 of Sep. 26, 1922 to G. S. Finney; **Can. Pat.** 232837 of Sep. 9, 1924 to Chromolithic Co.

192 [p. 633] **U. S. Pat.** 1582318 of Apr. 27, 1926 to Walter van de Mark.

193 [p. 633] **U. S. Pats.** 1677701 of Jul. 17, 1928 to W. H. Alton; 1931754 of Oct. 24, 1933 to P. S. Denning; 1954778 of Apr. 10, 1934 to E. H. Nichols; 2046295 of Jun. 30, 1936 to E. H. Nichols; 2054317 of Sep. 15, 1936 to H. R. Gundlach; 2070300 of Feb. 9, 1937 to P. R. Smith; 2070359 of Feb. 9, 1937 to C. E. Hillers; **Can. Pat.** 350356 of May 21, 1935 to E. H. Nichols; **Brit. Pat.** 440481 of Jan. 1, 1936 to P. E. McEllin.

194 [p. 633] **U. S. Pats.** 1766815 of Jun. 24, 1930 to H. C. Fisher; 1831469 of Nov. 10, 1931 to A. W. Sewall; 1831784 of Nov. 10, 1931 to G. H. Brown; 2004682 of Jun. 11, 1935 to S. G. Wright; 2010620 of Aug. 6, 1935 to S. G. Wright; 2033656 of Mar. 10, 1936 to P. R. Smith.

195 [p. 633] **U. S. Pat.** 1970471 of Aug. 14, 1934 to E. H. Nichols; 2061246 of Nov. 17, 1936 to E. H. Nichols; **Can. Pat.** 360470 of Sep. 8, 1936 to E. H. Nichols.

196 [p. 633] **U. S. Pats.** 1169506 of Jan. 25, 1916 to S. W. Osgood; 1788625 of Jan. 13, 1931 to H. L. Levin; 1944800 of Jan. 23, 1934 to E. H. Nichols; **Can. Pat.** 333086 of Jun. 6, 1933 to Patent & Licensing Corp.; 365590 of Apr. 20, 1937 to E. H. Nichols.

197 [p. 633] **U. S. Pats.** 2057677, 2057678 and 2057679 of Oct. 20, 1936 to H. R. Gundlach; **Can. Pats.** 364802, 364803, 364804 and 364806 of Mar. 16, 1937 to Central Commercial Co.

198 [p. 633] **U. S. Pats.** 1169506 of Jan. 25, 1916 to S. W. Osgood; 1572425 of Feb. 9, 1926 to H. C. Fisher; 1631628 of Jun. 7, 1927 to H. C. Fisher; 1742440 of Jan. 7, 1930 to H. C. Fisher; 1766814 of Jun. 24, 1930 to H. C. Fisher; 1766891 of Jun. 24, 1930 to H. C. Fisher; 1855210 and 1855211 of Apr. 26, 1932 to S. F. Walton; 1860040 of May 24, 1932 to Z. C. Loebel; 1876629 of Sep. 13, 1932 to P. S. Denning; 1898345 of Feb. 21, 1933 to P. S. Denning; 1921010 of Aug. 8, 1933 to H. L. Small; 1923835 of Aug. 22, 1933 to Z. C. Loebel; 1939930 of Dec. 19, 1933 to H. L. Small; 1970953 of Aug. 21, 1934 to O. R. Douthett; Reissue 19372 of Nov. 13, 1934 to S. F. Walton; 1991981 of Feb. 19, 1935 to C. E. Hillers; 2001448 of May 14, 1935 to M. R. Beasley; 2003849 and 2003850 of Jun. 4, 1935 to W. H. Alton; 2010619 of Aug. 6, 1935 to S. G. Wright; 2021716 of Nov. 19, 1935 to O. R. Douthett; 2062080 of Nov. 24, 1936 to S. G. Wright; 2069486 of Feb. 2, 1937 to P. S. Tilden; Reissue 20295 of Mar. 16, 1937 to H. C. Fisher; **Can. Pats.** 283297 of Sep. 11, 1928 to Richardson Co.; 338825 of Jan. 23, 1934 to S. F. Walton; 340802 of Apr. 10, 1934 to Patent & Licensing Corp.; 347890 of Jan. 29, 1935 to Patent & Licensing Corp.; 353448 of Oct. 8, 1935 to Building Products, Ltd.; 364805 of Mar. 16, 1937 to Central Commercial Co.

199 [p. 633] **U. S. Pats.** 1876630 of Sep. 13, 1932 to P. S. Denning; 1913666 and 1913667 of Jun. 13, 1933 to N. P. Harshberger.

200 [p. 633] **U. S. Pat.** 1802008 of Apr. 21, 1931 to Dozier Finley.

201 [p. 633] **U. S. Pat.** 1920931 of Aug. 1, 1933 to Dozier Finley.

202 [p. 633] **U. S. Pat.** 1789443 of Jan. 20, 1931 to H. L. Levin.

203 [p. 633] **U. S. Pat.** 1944294 of Jan. 23, 1934 to Poole Maynard.

204 [p. 633] **U. S. Pats.** 1766892 of Jun. 24, 1930 to H. C. Fisher; 1782648 of Nov. 25, 1930 to H. C. Fisher.

205 [p. 633] **Can. Pat.** 283296 of Sep. 11, 1928 to Richardson Co.

206 [p. 633] **U. S. Pat.** 1782649 of Nov. 25, 1930 to H. C. Fisher.

207 [p. 633] **U. S. Pats.** 1233501 of Jul. 17, 1917 to J. C. Pelton; 1680941 of Aug. 14, 1928 to H. C. Fisher; **Can. Pat.** 283295 of Sep. 11, 1928 to Richardson Co.; **Brit. Pat.** of 1868 (Oct. 24), 3249 to R. Ferguson and G. Ford.

208 [p. 633] **U. S. Pats.** 1867661 of Jul. 19, 1932 to J. V. Freeman; 1920773 of Aug. 1,

1933 to S. F. Walton; 1925065 of Aug. 29, 1933 to R. L. Atkinson; 2007742 of Jul. 9, 1935 to Leslie Brown; **Can. Pat.** 312675 of Jun. 23, 1931 to J. V. Freeman; 328954 of Dec. 27, 1932 to J. V. Freeman; 346367 of Nov. 27, 1934 to R. L. Atkinson. **Brit. Pat.** 352789 of Jan. 13, 1930 to R. L. Atkinson.

209 [p. 633] **U. S. Pat.** 1792058 of Feb. 10, 1931 to R. L. Atkinson; **Ger. Pat.** Appl. V-30716 of May 5, 1934 to Vereinigte Stahlwerke A.-G.; 630738 of Jun. 4, 1936 to Vereinigte Stahlwerke A.-G.

210 [p. 633] **U S. Pat.** 1943525 of Jan. 16, 1934 to H. R. Gundlach.

211 [p. 634] **U. S. Pat.** 1248170 of Nov. 27, 1917 to E. J. Schroder; **Can. Pat.** 327171 of Nov. 1, 1932 to Arthur Edge; **Brit. Pat.** 449585 of Jun. 29, 1936 to W. F. Edwardes.

212 [p. 634] **Ger. Pat.** 434188 of Feb. 18, 1925 to Walter Simon.

213 [p. 634] **U. S. Pats.** 1830489 of Nov. 3, 1931 to I. E. Sproat; 1838287 of Dec. 29, 1931 to P. R. Schreurs; 1944066 of Mar. 12, 1935 to P. S. Denning.

214 [p. 634] **Ger. Pat.** 582388 of May 22, 1931 to Ernst Täuber.

215 [p. 634] **U. S. Pat.** 1098723 of Jun. 2, 1914 to J. R. Hamlin.

216 [p. 634] **U. S. Pat.** 1841309 of Jan. 12, 1932 to R. T. Vanderbilt.

217 [p. 634] **U. S. Pat.** 1925282 of Sep. 5, 1933 to Thomas Robinson.

218 [p. 634] **U. S. Pats.** 81078 of Aug. 18, 1868 to Michael Ehret, Jr.; 293491 and 293492 of Feb. 12, 1884 to H. M. Miner; 304744 of Sep. 9, 1884 to H. M. Miner; 624976 of May 16, 1899 to R. J. Redick.

219 [p. 634] **U. S. Pats.** 130376 of Aug. 13, 1872 to Howard Kirk and James Winsmore, Jr.; 278722 of Jun. 5, 1883 to H. M. Miner.

220 [p. 634] **U. S. Pats.** 81078 of Aug. 18, 1868 to Michael Ehret, Jr.; 453979 of Jun. 9, 1891 to G. S. Lee.

221 [p. 634] **Brit. Pat.** 407460 of Nov. 16, 1932 to Hans Wiedemann.

222 [p. 634] **Ger. Pats.** Appl. A-10800 of Mar. 14, 1904 to A. W. Andernach (Rejected); Appl. E-18313 of Aug. 8, 1912 to Ludwig Esselborn (Rejected).

223 [p. 634] **U. S. Pats.** 753982 of Mar. 8, 1904 to S. R. Holland; 854740 of May 28, 1907 to A. G. Hennion; 1235270 of Jul. 31, 1917 to J. B. Wise.

224 [p. 634] **U. S. Pat.** 1203403 of Oct. 31, 1916 to Mathias Poulson; **Brit. Pat.** of 1884 (Mar. 14), 4897 to A. Davidson.

225 [p. 634] **U. S. Pats.** 311388 of Jan. 27, 1885 to R. H. Thompson; 674219 of May 14, 1901 to J. A. Scharwath; 1612885 of Jan. 4, 1927 to Thomas Robinson; 1932144 of Oct. 24, 1933 to C. J. Pater; **Brit. Pat.** of 1900 (Apr. 23), 7503 to K. T. Muir; **Ger. Pat.** 216603 of May 21, 1908 to C. F. Weber, A.-G.

226 [p. 634] **U. S. Pats.** 1318096 of Oct. 7, 1919 to W. F. McKay; 1365711 of Jan. 18, 1921 to W. F. McKay.

227 [p. 634] See Reference 401.

228 [p. 634] **Ger. Pat.** Appl. L-76677 of Nov. 7, 1929 to Kelman Lieber.

229 [p. 634] **U. S. Pats.** 1320502 of Nov. 4, 1919 to J. T. Simpson; 1492609 and 1492610 of May 6, 1924 to J. T. Simpson.

230 [p. 634] **U. S. Pat.** 1783839 of Dec. 2, 1930 to Henri Gauvin; **Can. Pat.** 306211 of Dec. 2, 1930 to Henri Gauvin; **Ger. Pats.** Designs 1393434, 1393435 and 1393436 of Apr. 18, 1936 to Emil Häfele.

231 [p. 634] **U. S. Pats.** 86380 of Feb. 2, 1869 to H. F. Evans; 91133 of Jun. 8, 1869 to James Howard; 95689 of Oct. 12, 1869 to James Howard; 99710 of Feb. 8, 1870 to John Roberts; 146105 of Dec. 30, 1873 to J. F. Stairs; 275841 of Apr. 17, 1883 to H. M. Miner; 285490 of Sep. 25, 1883 to Josiah Jowitt; 304457 of Sep. 2, 1884 to L. L. Sagendorph; 310544 of Jan. 6, 1885 to L. L. Sagendorph; 341587 of May 11, 1886 to William Macrone; 690348 and 690349 of Dec. 31, 1901 to C. S. Bird; 758132 of Apr. 26, 1904 to G. M. Gest; 1121728 of Dec. 22, 1914 to Olav Jacobsen, Steven Troy and Halfdan Kjeldesen; 1152136 of Aug. 31, 1915 to G. R. Wyman; 1184070 of May 23, 1916 to E. J. Cady; 1381967 of Jun. 21, 1921 to C. M. Clarke; **Can. Pats.** 22456 of Sep. 15, 1885 to L. L. Sagendorph; 23333 of Feb. 3, 1886 to L. L. Sagendorph; **Ger. Pats.** 656 of Jul. 26, 1877 to C. Homburg; 54224 of Mar. 7, 1890 to L. Jacobius & Söhne; 84288 of Aug. 26, 1894 to Richard Müller; 102819 of Jul. 24, 1897 to Stephan

Mattar; 103899 Jul. of 29, 1898 to Wilhelm Höpfner; 138171 of Nov. 27, 1901 to Max Alker and Walter Vinzelberg; 145586 of Oct. 18, 1902 to Hendrik de Clercq; 170063 of Apr. 1, 1905 to Schwarz'sche Maschinenfabrik und Eisengieszerei Emanuel Bergmann; 177610 of Jan. 20, 1906 to Johan de Clercq; 180957 of Oct. 27, 1905 to Bergische Dachpappen- und Teerprodukten-Fabrik Gottfr. Aug. Nebeling & Comp., G.m.b.H.; 203502 of May 29, 1907 to Otto Thümmel; 231273 of Feb. 27, 1909 to Heinrich Krause; 236847 of Jan. 22, 1910 to Ernst Mallmann; 238564 of May 11, 1910 to Ernst Mallmann; 263765 of Sep. 7, 1912 to Maschinenfabrik Calbe a.S., G.m.b.H.; Design. 738658 of Apr. 1, 1920 to Hermann Meyer; Design. 1288687 of Jan. 4, 1934 to C. G. Haubold, A.-G.

232 [p. 634] **U. S. Pats.** 1275216 of Aug. 13, 1918 to E. J. Cady; 1268430 of Jun. 4, 1918 to E. J. Cady; 1268446 of Jun. 4, 1918 to G. E. Ferguson; 1315763 of Sep. 9, 1919 to C. T. Dickey; 1331729 of Feb. 24, 1920 to J. D. Taylor; 1338624 of Apr. 27, 1920 to O. A. Heppes and C. E. Rahr; 1698886 of Jan. 15, 1929 to R. T. Johnston; 1736633 of Nov. 19, 1929 to K. H. Schutté; 1752972 of Apr. 1, 1930 to C. J. Beaver; 1842111 of Jan. 19, 1932 to C. J. Pater; 1845775 of Feb. 16, 1932 to Joseph Zavertnik, Jr. and A. A. MacCubbin; 1884486 of Oct. 25, 1932 to Joseph Zavertnik, Jr.; 1920541 of Aug. 1, 1933 to Joseph Zavertnik, Jr. and A. A. MacCubbin; 1958984 of May 15, 1934 to C. J. Beaver; **Can. Pat.** 259398 of Mar. 30, 1926 to Flintkote Co.; **Ger. Pats.** 534059 of Mar. 7, 1929 to W. T. Glover & Co., Ltd.; 577087 of Apr. 9, 1931 to S. D. Warren Co.; 597290 of Feb. 7, 1932 to W. T. Glover & Co., Ltd.

233 [p. 634] **U. S. Pats.** 1268446 of Jun. 4, 1918 to G. E. Ferguson; 1558549 of Oct. 27, 1925 to R. T. Johnston; 1587652 of Jun. 8, 1926 to R. T. Johnston; 1631826 of Jun. 7, 1927 to Lester Kirschbraun and C. E. Rahr; 1712770 of May 14, 1929 to H. C. Koch; 1751889 of Mar. 25, 1930 to C. R. Sculley; 1765777 of Jun. 24, 1930 to A. E. Schutté; **Can. Pats.** 244209 and 244210 of Nov. 4, 1924 to Flintkote Co. and R. T. Johnston; 295309 of Dec. 3, 1929 to C. R. Sculley; 333673 of Jul. 4, 1933 to J. J. Beaver.

234 [p. 634] **U. S. Pats.** 1751889 of Mar. 25, 1930 to C. R. Sculley; 1765777 of Jun. 24, 1930 to A. E. Schutté.

235 [p. 635] **U. S. Pats.** 111611 of Feb. 7, 1871 to J. M. Cobb; 1435810 of Nov. 14, 1922 to J. V. Cunha; 1589537 of Jun. 22, 1927 to N. C. Pike; 1636750 of Jul. 26, 1927 to J. E. Miller; 1754024 of Apr. 8, 1930 to C. L. Keller.

236 [p. 635] "Asphalt Emulsions in Paper Making," by Joseph Rossman, *Paper Trade J.*, **89**, 215 (1929); **U. S. Pats.** 103199 of May 17, 1870 to Samuel Kingan; 251410 of Dec. 27, 1881 to W. B. Carpenter; 253656 of Feb. 14, 1882 to S. M. Allen; 253840 of Feb. 21, 1882 to W. B. Carpenter; 254461 of Mar. 17, 1882 to J. D. Cheever; 334974 of Jan. 26, 1886 to A. A. Oliver; 337472 of Mar. 9, 1886 to S. M. Allen; 429999 of Jun. 10, 1890 to C. A. Catlin; 436733 of Sep. 16, 1890 to J. W. Easton; 452763 of May 19, 1891 to Frederick Salathé; 460056 of Sep. 22, 1891 to Ernst Fahrig; 568518 of Sep. 29, 1896 to Henry Carmichael; 636657 of Nov. 7, 1899 to Franz Gatzsche; 915860 of Mar. 23, 1909 to H. L. Hollister; 1044126 Nov. 12, 1912 to W. D. A. Bost; 1062686 of May 27, 1913 to J. H. Amies; 1156122 of Oct. 12, 1915 to J. C. Woodley; 1166166 of Dec. 28, 1915 to J. C. Woodley; 1177267 of Mar. 28, 1916 to R. P. Perry; 1201756 of Oct. 17, 1916 to R. P. Perry; 1204632 of Nov. 14, 1916 to J. C. Woodley and R. P. Perry; 1240524 of Sep. 18, 1917 to J. C. Woodley; 1241146 of Sep. 25, 1917 to R. P. Perry; 1288158 and 1288159 of Dec. 17, 1918 to R. P. Perry; 1293185 and 1293186 of Feb. 4, 1919 to R. P. Perry and J. M. Jack; 1296114 of Mar. 4, 1919 to R. P. Perry; 1302209 of Apr. 29, 1919 to R. P. Perry; 1305081 of May 27, 1919 to J. P. Elliott; 1305125 of May 27, 1919 to J. J. Laing and C. W. Boling; 1305404 of Jun. 3, 1919 to R. P. Perry; 1307750 of Jun. 24, 1919 to R. P. Perry; 1316591 of Sep. 23, 1919 to R. P. Perry; 1321669 of Nov. 11, 1919 to L. O. Pickett; 1338037 of Apr. 27, 1920 to R. P. Perry; 1340181 of May 18, 1920 to G. L. Oliensis and J. S. Miller, Jr.; 1344620 of Jun. 29, 1920 to F. B. Davidson; 1352796 of Sep. 14, 1920 to J. P. Elliott; 1360313 of Nov. 30, 1920 to R. P. Perry; 1360684 of Nov. 30, 1920 to L. C. Roberts; 1365711 of Jan. 18, 1921 to W. F. McKay; 1379590 of May 24, 1921 to A. L. Clapp; 1382740 of Jun. 28, 1921 to R. P. Perry; 1386276 and 1386277 of Aug. 2, 1921 to R. P. Perry; 1387219 of Aug. 9, 1921 to H. F. Weiss; 1400312 of Dec. 13, 1921 to R. P. Perry; 1406174 of Feb. 14, 1922 to J. P. Elliott; 1411330 of Apr. 4, 1922 to J. P. Elliott; Reissue 15461 of Sep. 26, 1922 to R. P. Perry; 1432742 of Oct. 24, 1922 to F. J.

Commin; 1438966 of Dec. 19, 1922 to R. P. Perry; 1449221 of Mar. 20, 1923 to G. H. Ellis; 1467594 of Sep. 11, 1923 to H. F. Weiss; 1476570 of Dec. 4, 1923 to F. W. Adams; 1490362 of Apr. 15, 1924 to A. L. Clapp; 1498401 of Jun. 17, 1924 to R. P. Perry; 1499291 of Jun. 24, 1924 to H. M. Castner; 1511475 of Oct. 14, 1924 to K. B. Howell and C. R. Eckert; 1511949 of Oct. 14, 1924 to F. J. Commin; 1577074 of Mar. 16, 1926 to R. P. Perry and F. W. Adams; 1698298 of Jan. 8, 1929 to A. L. Clapp; 1698733 of Jan. 15, 1929 to R. P. Perry; 1708927 of Apr. 9, 1929 to Lester Kirschbraun; 1710320 of Apr. 23, 1929 to R. P. Perry; 1712002 of May 7, 1929 to J. A. Heany; 1753690 of Apr. 8, 1930 to G. A. Brown; 1769513 of Jul. 1, 1930 to A. M. Hinkson; 1771150 of Jul. 22, 1930 to E. P. Stevenson and H. A. Buron; 1778147 of Oct. 14, 1930 to E. S. Edwards; 1792098 of Feb. 10, 1931 to C. L. Keller; 1809316 of Jun. 9, 1931 to D. F. Smith, E. J. Pieper and C. C. Vogt; 1824430 of Sep. 22, 1931 to C. C. Hall; 1825869 of Oct. 6, 1931 to C. L. Keller; 1827700 and 1827701 of Oct. 13, 1931 to Lester Kirschbraun; 1844951 of Feb. 16, 1932 to H. C. Fisher, A. M. Overton and C. L. Keller; 1859414 of May 24, 1932 to E. P. Stevenson; 1875018 of Aug. 30, 1932 to M. H. Kliefoth; 1877377 of Sep. 13, 1932 to Gibson Yungblut; 1878300 of Sep. 20, 1932 to E. P. Stevenson and H. A. Buron; 1900699 of Mar. 7, 1933 to G. H. Ellis; 1901382 of Mar. 14, 1933 to E. P. Stevenson; 1901930 of Mar. 21, 1933 to E. J. Pieper, D. F. Smith and C. C. Vogt; 1923888 of Aug. 22, 1933 to Thomas Robinson; 1924601 of Aug. 29, 1933 to H. C. Fisher; 1949249 of Feb. 27, 1934 to H. C. Fisher; 1953397 of Apr. 3, 1934 to E. J. Eimer; 1970426 of Aug. 14, 1934 to H. L. Levin; 1981573 of Nov. 20, 1934 to Earl Stafford; 2022311 of Nov. 26, 1935 to H. C. Fisher; 2023675 of Dec. 10, 1935 to H. C. Fisher; 2036466 of Apr. 7, 1936 to G. H. Ellis; **Can. Pats.** 245711, 245713 and 245714 of Dec. 30, 1924 to Lester Kirschbraun; 262825 of Jul. 20, 1926 to Richardson Co.; 270963 of May 24, 1927 to A. L. Clapp; 281701 of May 16, 1928 to Herbert Abraham; 291275 of Jul. 9, 1929 to World Bestos Corp.; 303662 of Sep. 2, 1930 to Richardson Co.; 319982 of Feb. 23, 1932 to Patent & Licensing Corp.; 326380 of Sep. 27, 1932 to Flintkote Corp.; 333441 and 333442 of Jun. 20, 1933 to Richardson Co.; 343186 of Jul. 17, 1934 to International Bitumen Emulsions Corp.; **Brit. Pats.** of 1841 (Dec. 16), 9189 to J. A. Fanshawe; of 1860 (Aug. 2), 1685 to Abraham Ripley; of 1862 (Jun. 17), 1788 to William Sinnock and John Rowley; of 1863 (Jun. 25), 1603 to William Kirrage; of 1864 (Apr. 28), 1072 to T. G. Ghislin; of 1870 (Oct. 27), 2831 to J. G. Willans; of 1872 (Apr. 27), 1264 to W. A. Lyttle; of 1876 (Mar. 27), 1309 to Caleb Tayler; of 1876 (Apr. 26), 1750 to J. Noad; of 1888 (May 30), 7933 to O. Imray; of 1893 (Mar. 28), 6565 to Siemens Cros & Co. and E. F. A. Obach; of 1897 (Apr. 23), 10181 to M. van Look; of 1898 (Mar. 30), 7678 to Ludwig Hatschek; of 1909 (Apr. 27), 9929 to J. H. Amies; of 1911 (Apr. 6), 8622 to W. H. Rymer; of 1915 (Jun. 15), 8815 to J. C. Woodley; 102826 of Jan. 7, 1916 to J. E. Clark; 125492 of Apr. 12, 1918 to S. G. Kelsey; 157217 of Mar. 23, 1922 to Max Rogler; 165240 of Apr. 1, 1920 to Harold Wade; 166886 of Jul. 7, 1921 to Harold Wade; 167334 of Aug. 10, 1920 to G. L. Oliensis and J. S. Miller, Jr.; 174352 of Jan. 18, 1922 to Barrett Co.; 174668 of Jan. 31, 1922 to T. K. Webster; 341542 of Nov. 1, 1928 to Richardson Co.; 354001 of Jul. 13, 1929 to H. L. Becher; 383636 of May 16, 1931 to Richardson Co.; 386991 of May 16, 1931 to Richardson Co.; 388643 of Jun. 27, 1932 to F. B. Dehn; **Ger. Pats.** 179577 of Jun. 10, 1904 to J. B. Granjon and J. F. J. Berchet; 253377 of Jul. 19, 1911 to Albin Baer; 337769 of Jan. 30, 1920 to Max Rogler; 376742 of Mar. 21, 1921 to F. J. Commin; 514730 of Nov. 10, 1925 to Albert Sommer; 591112 of Dec. 28, 1933 to Richardson Co.; 602292 of May 10, 1931 to Richardson Co.; 602312 of Jan. 10, 1931 to C. A. Braun; 602616 of Oct. 16, 1932 to C. A. Braun; Appl. W-46681 of Jul. 3, 1915 to J. C. Woodley (withdrawn); 613620 of Mar. 27, 1929 to International Bitumen Emulsions Corp.; 625123 of Dec. 14, 1929 to Bitumen Handelsgesellschaft m.b.H.; **Fr. Pats.** 683976 of Oct. 26, 1929 to Richardson Co.; 714517 of Mar. 17, 1931 to Insulite Co.; **Hungarian Pat.** 100522 of Aug. 7, 1929 to Albert Sommer.

237 [p. 635] **U. S. Pat.** 1925584 of Sep. 5, 1933 to H. C. Fisher.

238 [p. 635] **Ger. Pat.** 101435 of Feb. 24, 1898 to Ludwig Hatschek.

239 [p. 639] "Waterproof Paper or Board," by Lester Kirschbraun; *Paper Trade J.*, **74**, 48 (1922); "Asphalt Emulsions in Paper Making," by Joseph Rossman, *Paper Trade J.*, **89**, 215 (1929); "Asphalt Board," by D. P. Bailer, *Paper Mill*, **53**, 10, 24 and 26 (1930); Nach

neueren Verfahren mit Asphaltemulsionen in Stoff imprägnierte Pappen, Papiere und Formkörper aus Papierstoff für elektrotechnische und ähnliche Zwecke," by Fritz Hoyer, *Asphalt u. Teer*, **31**, 1034 (1931); "Die Anwendung der KB-Emulsion in der Papierindustrie," by W. H. Wobbe, *Papierfabrikant*, **29**, 333 (1931); "Asphaltpapiere nach neuen Herstellungsverfahren," by Fritz Hoyer, *Kunstoffe*, **22**, 11 (1932); "Mit Asphaltemulsionen imprägnierte Sackpapiere," by Fritz Hoyer, *Asphalt u. Teer*, **34**, 965 (1934); "Wasserdichte Wellpappen und Wellpappenpackungen," by Fritz Hoyer, *Papierfabrikant*, **32**, 419 (1934); "Bituminemulsionen in Papieren und Pappen," by H. Calmels, *La Papetreie*, **56**, 58 (1934); **U. S. Pats.** 577135 of Feb. 16, 1897 to P. H. Holmes; 1021660 of Mar. 26, 1912 to A. L. Clapp; 1191932 of Jul. 18, 1916 to J. C. Loyer and O. M. Loyer; 1228580 of Jun. 5, 1917 to G. W. Miles; 1234315 of Jul. 24, 1917 to E. B. Eising; Reissue 14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1302810 of May 6, 1919 to Lester Kirschbraun; 1330443 of Feb. 10, 1920 to G. C. Glynn; 1401791 of Dec. 27, 1921 to Lester Kirschbraun; 1417839 of May 30, 1922 to Lester Kirschbraun; 1461445 of Jul. 10, 1923 to Lester Kirschbraun; 1479044 of Jan. 1, 1924 to Lester Kirschbraun; 1511949 of Oct. 14, 1924 to F. J. Commin; 1515821 of Nov. 18, 1924 to H. C. Avery; 1526522 of Feb. 17, 1925 to Lester Kirschbraun; 1526553 of Feb. 17, 1925 to Lester Kirschbraun and T. W. Morse; 1533100 of Apr. 14, 1925 to A. L. Clapp; 1536399 of May 5, 1925 to Lester Kirschbraun; 1537949 and 1537950 of May 19, 1925 to Lester Kirschbraun; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; 1549992 of Aug. 18, 1925 to Lester Kirschbraun; 1561728 of Nov. 17, 1925 to Lester Kirschbraun; 1563642 of Dec. 1, 1925 to T. W. Morse; 1592294 of Jul. 13, 1926 to A. L. Clapp; 1606427 and 1606428 of Nov. 9, 1926 to Lester Kirschbraun; 1615303 of Jan. 25, 1927 to Lester Kirschbraun; 1616901, 1616902 and 1616903 of Feb. 8, 1927 to Lester Kirschbraun; 1616921 of Feb. 8, 1927 to C. E. Rahr and Lester Kirschbraun; 1621791 of Mar. 22, 1927 to Lester Kirschbraun; 1657585 of Jan. 31, 1928 to R. P. Perry; 1659401 of Feb. 14, 1928 to Lester Kirschbraun; 1663095 of Mar. 20, 1928 to R. P. Perry; 1668760 of May 8, 1928 to A. L. Clapp; 1670844 of May 22, 1928 to R. R. Cone; 1672262 of Jun. 5, 1928 to Lester Kirschbraun; 1686818 of Oct. 9, 1928 to Lester Kirschbraun; 1691752 of Nov. 13, 1928 to Edmund Bache; 1700561 of Jan. 29, 1929 to F. J. Commin and A. H. J. Wright; 1707491 of Apr. 2, 1929 to R. P. Perry; 1708926 of Apr. 9, 1929 to Lester Kirschbraun; 1722431, 1722432 and 1722434 of Jul. 30, 1929 to Lester Kirschbraun; 1723355 of Aug. 6, 1929 to Lester Kirschbraun; 1723361 of Aug. 6, 1929 to H. L. Levin; 1725645, 1725646 and 1725647 of Aug. 20, 1929 to Lester Kirschbraun; 1727003 of Sep. 3, 1929 to A. L. Clapp; 1735030 of Nov. 12, 1929 to Edmund Bache; 1738509 of Dec. 3, 1929 to Lester Kirschbraun; 1747232 of Feb. 18, 1930 to Dozier Finley; 1767532 of Jun. 24, 1930 to Lester Kirschbraun; 1771744 and 1771745 of Jul. 29, 1930 to A. L. Clapp; 1777447 of Oct. 7, 1930 to Frederic Queck; 1781645 of Nov. 11, 1930 to Lester Kirschbraun; 1785357 of Dec. 16, 1930 to H. L. Levin; 1786270 of Dec. 23, 1930 to H. M. Spencer; 1825422 of Sep. 29, 1931 to R. P. Rose; 1827700 of Oct. 13, 1931 to Lester Kirschbraun; 1856946 of May 3, 1932 to W. A. Darrah; 1884761 of Oct. 25, 1932 to H. L. Levin; 1889642 and 1889643 of Nov. 29, 1932 to G. F. Davis; 1905212 of Apr. 25, 1933 to C. A. Braun; 1912511 of Jun. 6, 1933 to F. L. Carson; 1918462 of Jul. 18, 1933 to Konrad Erdmann; 1926982 of Sep. 12, 1933 to Lester Kirschbraun; 1927047 of Sep. 19, 1933 to Thomas Robinson; 1958020 of May 8, 1934 to Thomas Robinson; 2016568 of Oct. 8, 1935 to P. R. Zinser; 2026594 of Jan. 7, 1936 to H. W. Richter; 2045410 of Jun. 23, 1936 to G. A. Richter and M. O. Schur; 2057331 of Oct. 13, 1936 to H. C. Fisher and George Acus; **Can. Pats.** 203698 of Sep. 7, 1920 to Lester Kirschbraun; 226168 of Nov. 21, 1922 to Lester Kirschbraun; 229000 of Feb. 27, 1923 to F. J. Commin; 243952 of Oct. 28, 1924 to Lester Kirschbraun; 245539 of Dec. 23, 1924 to G. J. Manson; 245711, 245713 and 245714 of Dec. 30, 1924 to Lester Kirschbraun; 245877 of Jan. 6, 1925 to Lester Kirschbraun; 246220 of Jan. 20, 1925 to Flintkote Co.; 248144 of Mar. 31, 1925 to Lester Kirschbraun; 248219 of Mar. 31, 1925 to Lester Kirschbraun; 284540 of Apr. 7, 1925 to Flintkote Co.; 251518 of Jul. 7, 1925 to Flintkote Co.; 256080 of Dec. 8, 1925 to Lester Kirschbraun; 260604 of May 18, 1926 to Lester Kirschbraun; 264914 of Oct. 12, 1926 to Lester Kirschbraun; 276092 of Dec. 6, 1927 to Lester Kirschbraun; 281128 of Jun. 19, 1928 to Flintkote Co., 297615 of Feb. 18, 1930 to Flintkote Co.; 305679 of Nov. 11, 1930 to Flintkote Co.; 325757 of Sep. 6, 1932 to International Paper Co.; 340620 of Apr. 3, 1934 to

Standard Oil Development Co.; 340781 of Apr. 10, 1934 to International Bitumen Emulsions Corp.; 354906 of Dec. 24, 1935 to Barrett Co.; **Brit. Pats.** of 1910 (May 4), 15711 to Friedrich Raschig; 151029 of Mar. 6, 1919 to Lester Kirschbraun; 162727 of May 2, 1921 to F. J. Commin; 167613 of Aug. 18, 1921 to F. J. Commin; 174114 of Sep. 7, 1920 to Lester Kisrchbraun; 185816 of Jun. 6, 1921 to Lester Kirschbraun; 262828 of Dec. 14, 1926 to Hans Friedländer; 273281 of Jun. 16, 1927 to N. V. de Bataafsche Petroleum Maatschappij; 276395 of Apr. 19, 1926 to J. J. H. Sturmey; 359902 and 359950 of Jul. 21, 1930 to H. D. Elkington; 366521 of Oct. 30, 1930 to H. D. Elkington; 403116 of Jun. 8, 1932 to F. B. Dehn; 418253 of Sep. 13, 1933 to International Bitumen Emulsions Corp.; and A. G. Bloxam; 442045 of Jan. 23, 1936 to Cellufoam Corp.; **Ger. Pats.** 2041 of Nov. 16, 1877 to F. Leroy; 216753 of Sep. 6, 1906 to Julius Kathe; 296124 of Aug. 10, 1915 to Werner Schmidt and Emil Heuser; 305525 of Jul. 12, 1917 to Werner Schmidt and Emil Heuser; 321232 of Mar. 15, 1918 to Werner Schmidt and Emil Heuser; 328733 of Nov. 9, 1918 to Wäschefabrik Gebr. Simion, A.-G.; 337769 of Jan. 30, 1920 to Max Rogler; 352860 of Nov. 21, 1920 to Farbwerke vorm. Meister, Lucius & Brüning; 392901 of Mar. 27, 1921 to I. G. Farbenindustrie, A.-G.; 392902 of Aug. 6, 1921 to I. G. Farbenindustrie, A.-G.; 401546 of Jun. 2, 1921 to Lester Kirschbraun; 404070 of Nov. 2, 1917 to Eduard Dyckerhoff; 442010 of Jun. 13, 1924 to Lester Kirschbraun; 447505 of Jun. 11, 1924 to Lester Kirschbraun; 536019 of Sep. 14, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 542342 of Feb. 24, 1930 to Deutsche Heraklith, A.-G.; 542440 of Aug. 20, 1929 to Max Tausent and Hans Eggert; 569463 of Sep. 2, 1933 to Herman Doehler; Appl. B-140683 of Nov. 29, 1928 to International Bitumen Emulsions Corp.; Appl. F-130.30 of Oct. 28, 1930 to Flintkote Co.; Appl. J-42125 of Jul. 21, 1931 to I. G. Farbenindustrie, A.-G.; 614035 of Apr. 8, 1928 to International Bitumen Emulsions Corp.; 616179 of Jul. 22, 1935 to Chem. Fabrik Flörsheim, Dr. H. Noerdlinger, A.-G.; 625123 of Feb. 4, 1936 to Bitumen Handelsgesellschaft m.b.H.; **Fr. Pats.** 526335 of Oct. 13, 1920 to Lester Kirschbraun; 529295 of Dec. 31, 1920 to F. J. Commin; 735847 of Apr. 22, 1932 to Bitumen Investments, Inc.; 763131 of Apr. 24, 1934 to Bitumen Investments, Inc.; **Swiss Pat.** 91579 of Mar. 31, 1919 to Lester Kirschbraun; **Austrian Pat.** 140196 of Jan. 10, 1935 to Johann Fröhlich (Firma); **Danish Pat.** 52501 of Dec. 28, 1936 to M. B. C. Emulsion Comp. A/S.

240 [p. 635] **U. S. Pats.** 103536 of May 31, 1870 to T. R. Abbot; 111611 of Feb. 7, 1871 to J. M. Cobb; 119059 of Sep. 18, 1871 to H. N. Stimson; 151683 of Jun. 9, 1874 to Elias Burnham; 230148 of Jul. 20, 1880 to G. S. Page; 825744 of Jul. 10, 1906 to A. E. Millington; 1374885 of Apr. 12, 1921 to C. S. Hathaway; **Brit. Pats.** of 1838 (Jul. 11), 7731 to John Bethell; of 1847 (Nov. 25), 11979 to William Hutchison; of 1855 (Apr. 27), 948 to R. P. Coignet; of 1869 (Jun. 29), 275 to N. C. Szerelmey; of 1873 (Apr. 25), 1499 to Peter Jensen; of 1874 (Aug. 7), 2732 to W. E. Gedge; of 1876 (Jul. 27), 3028 to G. H. Hebblethwaite; of 1880 (Oct. 9), 4107 to J. C. Mewburn; **Ger. Pats.** 656 of Jul. 26, 1877 to C. Homberg; 29444 of Jun. 17, 1884 to E. P. Louvot; 54224 of Mar. 7, 1890 to L. Jacobus & Sons; 84288 of Aug. 26, 1894 to Richard Müller; 102819 of Jul. 24, 1897 to Stephan Mattar; 131171 of Nov. 27, 1901 to Max Alker and Walter Vinzelberg; 170063 of Apr. 1, 1905 to Schwarz'che Maschinenfabrik; 177610 of Jan. 20, 1906 to Johan de Clercq; 180958 of Oct. 27, 1906 to Bergische Dachpappen- und Teerprodukten-Fabrik; 236847 of Jan. 22, 1907 to Ernst Mallmann; 238564 of Mar. 11, 1910 to Ernst Mallmann; 263765 of Sep. 7, 1912 to Maschinenfabrik Calbe, a.S., G.m.b.H.; 284886 of Oct. 23, 1913 to Schatz & Hübner.

241 [p. 635] **U. S. Pats.** 820694 of May 15, 1906 to L. A. Bond; 876008 of Jan. 7, 1908 to F. C. Overbury; 876010 of Jan. 7, 1908 to F. C. Overbury; **Can. Pats.** 263348 of Aug. 10, 10, 1926 to W. R. Seigle; **Brit. Pats.** of 1843 (Oct. 5), 9890 to Benedict Albino; of 1867 (Oct. 19), 2946 to J. Anderson; of 1876 (May 21), 2295 to J. S. Norrie; of 1887 (Sep. 22), 12864 to J. C. Lyman; of 1892 (Apr. 23), 7698 to A. J. Boult; of 1893 (Jul. 28), 14551 to G. McTear; 308890 of Mar. 19, 1928 to Eikichi Sakuma; **Ger. Pats.** 122893 of May 5, 1899 to A. W. Andernach; 203502 of May 29, 1907 to Otto Thümmel; 208378 of Sep. 13, 1905 to Otto Schreiber; 276619 of Dec. 14, 1913 to Anhydat-Lederwerke, a.G.; 295863 of Apr. 16, 1916 to Benno Schilde and Adolf Boleg; 231273 of Feb. 27, 1909 to Heinrich Krause; Design 738658 of Apr. 1, 1920 to Hermann Meyer.

242 [p. 637] **U. S. Pat.** 1965703 of Jul. 10, 1934 to E. H. Hyde.

243 [p. 638] **U. S. Pats.** 1312682 of Aug. 12, 1919 to A. L. Clapp; 1827026 of Oct. 13, 1931 to A. G. Leonard, Jr.; 1975584 of Oct. 2, 1934 to A. G. Leonard, Jr.; **Ger. Pat.** 316540 of Jun. 9, 1915 to Theodor Horst and Hermann Stöcker.

244 [p. 638] **U. S. Pats.** 1589537 of Jun. 22, 1926 to A. R. Lukens and N. C. Pike; 2044012 of Jun. 16, 1936 to I. J. Novak; 2049469 of Aug. 4, 1936 to I. J. Novak; **Can. Pat.** 345218 of Oct. 9, 1934 to Raybestos-Manhattan, Inc.

245 [p. 638] **U. S. Pats.** 1381967 of Jun. 21, 1921 to C. M. Clarke; 1389630 of Sep. 6, 1921 to C. A. Cooney; 1508959 of Sep. 16, 1924 to H. A. Cumfer; 1511187 of Oct. 7, 1924 to George Ritter; 1579003 of Mar. 30, 1926 to H. C. Koch; 1588748 of Jun. 15, 1926 to H. C. Koch; 1603976 of Oct. 19, 1926 to H. M. Nicholls; 1712770 of May 14, 1929 to H. C. Koch.

246 [p. 638] **U. S. Pats.** 111611 of Feb. 7, 1871 to J. M. Cobb; 349463 of Sep. 21, 1886 to E. G. Sparks; 362547 of May 10, 1887 to Isaac Sherck and Joseph Batig, Jr.; 380915 of Apr. 10, 1888 to H. M. Miner; 1417841 of May 30, 1922 to Lester Kirschbraun; 1345810 of Nov. 14, 1922 to J. V. Cunha; 1636750 of Jul. 26, 1927 to J. E. Miller; 1754024 of Apr. 8, 1930 to C. L. Keller; 1760606 of May 27, 1930 to N. C. Pike; 1791040 of Feb. 3, 1931 to W. H. Richardson; 1816596 of Jul 28, 1931 to Robert Maclean; 1982679 of Dec. 4, 1934 to Robert Maclean.

247 [p. 638] **U. S. Pats.** 349463 of Sep. 21, 1886 to E. G. Sparks; 1277986 of Sep. 3, 1918 to W. C. Merrill.

248 [p. 638] **U. S. Pats.** 1268430 of Jun. 4, 1918 to E. J. Cady; 1854191 and 1854192 of Apr. 19, 1932 to H. E. Holcomb.

249 [p. 638] **U. S. Pat.** 1762329 of Jun. 10, 1929 to W. G. Dudleston; **Can. Pat.** 356342 of Mar. 3, 1936 to T. P. Bishop.

250 [p. 638] **U. S. Pats.** 1762336 of Jun. 10, 1929 to Lester Kirschbraun; 1776590 of Sep. 23, 1930 to Lester Kirschbraun; 1930646 of Oct. 17, 1933 to P. P. Gray and E. E. Werle; 2050483 of Aug. 11, 1936 to H. D. Brown; **Can. Pats.** 299376 and 299377 of Apr. 15, 1930 to Patent & Licensing Corp.

251 [p. 638] **U. S. Pat.** 1776586 of Sep. 23, 1930 to G. P. Heppes; 2023019 of Dec. 3, 1930 to G. P. Heppes; 2040514 of May 12, 1936 to J. F. Dillon; 2040529 of May 12, 1936 to J. W. Pearl; **Can. Pats.** 359224 and 359225 of Jul. 21, 1936 to Barrett Co.

252 [p. 638] **U. S. Pat.** 1331365 of Feb. 17, 1920 to R. P. Perry.

253 [p. 638] **U. S. Pats.** 1414462 of May 2, 1922 to L. T. Frederick; 1765778 of Jun. 24, 1930 to A. E. Schutte.

254 [p. 638] **U. S. Pats.** 1286057 of Nov. 26, 1918 to R. W. E. Moore; 1831630 of Nov. 10, 1931 to F. B. Manker.

255 [p. 638] **U. S. Pat.** 2062382 of Dec. 1, 1936 to J. H. Weller.

256 [p. 638] **U. S. Pats.** 1369186 of Feb. 22, 1921 to R. P. Perry; 1471980 of Oct. 23, 1923 to A. J. Ryan; 1715392 of Jun. 4, 1929 to A. E. Schutte; 1778733 of Oct. 21, 1930 to H. A. Sjodahl; **Can. Pat.** 306122 of Nov. 25, 1930 to Patent & Licensing Corp.

257 [p. 638] **U. S. Pat.** 230148 of Jul. 20, 1880 to G. S. Page; **Ger. Pat.** 112629 of May 5, 1899 to A. W. Andernach; **Fr. Pat.** 343704 of Jun. 4, 1904 to J. B. Granjon and J. F. J. Berchet.

258 [p. 638] **U. S. Pat.** 1381967 of Jun. 21, 1921 to C. M. Clark; 2023019 of Dec. 3, 1935 to G. P. Heppes.

259 p. 638] **U. S. Pats.** 1673186 of Jun. 12, 1928 to H. A. Cumfer; 1685078 of Sep. 25, 1928 to F. W. Adams; **Can. Pat.** 244602 of Nov. 18, 1924 to Guyton & Cumfer Mfg. Co.

260 [p. 638] **U. S. Pats.** 1383726 of Jul. 5, 1921 to Otterbein Kiracofe; 1547129 of Jul. 21, 1925 to George Ritter; 1685078 of Sep. 25, 1928 to F. W. Adams; **Brit. Pat.** 340749 of Dec. 13, 1929 to E. Rudin and Heinrich Kollbrunner.

261 [p. 638] **U. S. Pat.** 1437312 of Nov. 28, 1922 to R. G. Jackson.

262 [p. 638] **U. S. Pats.** 1443971 of Feb. 6, 1923 to Alfred Anderson; 1443975 of Feb. 6, 1923 to T. F. Caldwell.

263 [p. 638] **U. S. Pat.** 1942763 of Jan. 9, 1934 to W. R. Menshon; **Can. Pat.** 336924 of Nov. 7, 1933 to Barrett Co.; 363985 of Feb. 9, 1937 to Certainteed Products Corp.

264 [p. 638] **U. S. Pat.** 1845299 of Feb. 16, 1932 to E. A. Leonard.

265 [p. 638] **U. S. Pat.** 1870564 of Aug. 9, 1932 to H. P. Hayden.

266 [p. 639] DIN-4031, Wasserdruckhaltende Dichtungen aus nackten Teerpappen oder nackten Asphalt-Bitumenpappen für Bauwerke, (B 1/2), Jan. 30, 1921; DVM-2117, Rohdachpappe, (B 1/2, M 2/2), Sep. 26, 1930; DVM-2118, Prüfung von Rohdachpappe, (B 1/2, M 2/2), Jan. 30, 1930; DVM-2119, Wollfilzpappe, (B 1/2, M 2/2), Jan. 30, 1931; DVM-2120, Prüfung von Wollfilzpappen, (B 1/2, M 2/2); DVM-2121, Teerdachpappen, beiderseitig besandet, (B 1/2, M 2/2), Apr. 1928; DVM-2122, Tränkmassen für besandete Teerdachpappen, (B 1/2, M 2/2), Apr. 1928; DVM-2123, Prüfung von Teerdachpappe, (B 1/2, M 2/2), Apr. 1928; DVM-2124, Prüfung von Tränkmassen für Teerpappen, (B 1/2, M 2/2), Apr. 1928; DVM-2125, Teerdachpappen, einerseitig besandet, (B 1/2, M 2/2), Jul. 1929; DVM-2126, Nackte Teerpappen, (B 1/2, M 2/2), Jul. 1929; DVM-2127, Tränkmassen für nackte Teerpappen, (B 1/2, M 2/2), Jul. 1929; DVM-2128, Asphaltbitumen-Dachpappen (teerfrei) mit beiderseitiger Asphaltbitumendeckschicht, (B 1/2, M 2/2); DVM-2129, Tränkmassen für Asphaltbitumen-Dachpappen, (B 1/2, M 2/2); DVM-2130, Prüfung von Asphaltbitumen-Dachpappen (teerfrei) mit beiderseitiger Asphaltbitumendeckschicht, (B 1/2, M 2/2); DVM-2131, Prüfung von Tränkmassen für Asphaltbitumen-Dachpappen, (B 1/2, M 2/2); DVM-2136, Dachanstrichstoffe, Steinkohlenteere, (B 1/2, M 2/2), Sep. 1930 (new); DVM-2137, Prüfung von Steinkohlenteer als Dachanstrichstoffe, (B 1/2, M 2/2), Sep. 1930 (new); DVM-2138, Klebemassen für Dachpappe, Steinkohlenteererzeugnisse, (B 1/2, M 2/2), Sep. 1930 (new); DVM-2139, Prüfung von Steinkohlenteererzeugnissen als Klebemassen für Dachpappe, (B 1/2, M 2/2), Sep. 1930 (new); all the foregoing issued by Deutscher Verband für die Materialprüfungen der Technik, published by Beuth-Verlag, Berlin.

267 [p. 639] **U. S. Pat.** 1577890 of Mar. 23, 1926 to C. D. Abraham.

268 [p. 640] **U. S. Pat.** 1729681 of Oct. 1, 1929 to C. H. Panthen.

269 [p. 640] **U. S. Pats.** 414247 of Nov. 5, 1889 to George Phillips; 758841 of May 3, 1904 to C. W. Kennon; 1981664 of Nov. 20, 1934 to D. A. Quarles.

270 [p. 640] **U. S. Pat.** 528019 of Jun. 5, 1894 to E. M. Caffall.

271 [p. 640] **U. S. Pats.** 526552 of Sep. 25, 1894 to Charles Arnoudts; 1512659 and 1512660 of Oct. 21, 1924 to C. L. Wright and Edward Toal.

272 [p. 640] **U. S. Pats.** 546960 of Sep. 24, 1895 to F. A. Lorbert; 686282 of Nov. 12, 1901 to Edward Gold.

273 [p. 640] "The Water-resistance of Treated Canvas During Continuous Exposure to the Weather," by F. P. Veitch and T. D. Jarrell, *J. Ind. Eng. Chem.*, 13, 672 (1921).

274 [p. 640] "Effects of Treating Materials and Outdoor Exposure upon the Water-resistance and Tensile Strength of Cotton Duck," by T. D. Jarrell and H. P. Holman, *J. Ind. Eng. Chem.*, 15, 607 (1923).

275 [p. 640] "Waterproofing and Mildewproofing Cotton Duck," by H. P. Holman, B. S. Levine and T. D. Jarrell, Farmers' Bull. No. 1157, U. S. Dept. Agri., Washington, D. C. (Oct. 1920); **Ger. Pat.** 343164 of Jun. 26, 1929 to O. Röszler.

276 [p. 640] **Ger. Pat.** 127852 of Jan. 18, 1902 to Carl Baswitz.

277 [p. 640] **U. S. Pat.** 1618416 of Feb. 22, 1927 to T. H. Fairbrother and Arnold Renshaw.

278 [p. 640] **U. S. Pat.** 1364475 of Jan. 4, 1921 to G. C. Bailey and Felix Boettner.

279 [p. 640] **Ger. Pat.** 391148 of Jan. 26, 1923 to Warnecke & Böhm, A.-G.

280 [p. 640] "Cadmium Oleate: A New Impregnating Agent," by A. H. Grimshaw, *Chem. Zentr.*, II, 2326 (1930).

281 [p. 640] **U. S. Pat.** 1026855 of May 21, 1912 to Carleton Ellis.

282 [p. 640] "Preservation of Textile Fish Nets," by A. C. Robertson, *J. Ind. Eng. Chem.*, 23, 1093 (1931).

283 [p. 640] "Untersuchungen über Ursachen der Zerstörung von Grundwasser- Isoliermaterialien," by Carl Alfeis, Druckerei Gutenberg, Braunschweig (1931); **U. S. Pats.** 38646 of May 26, 1863 to D M. Ayer; 731002 of Jun. 16, 1903 to James Williams; **Ger. Pats.** 226975 of May 8, 1908 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger; A.-G.; 232380 of May 23, 1908 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger; A.-G.

284 [p. 640] **U. S. Pat.** 1745134 of Jan. 28, 1930 to C. B. White and Eugene Schaefer.

285 [p. 640] **Can. Pat.** 301858 of Jul. 8, 1930 to British Cotton Industry Research Association.

286 [p. 641] "Bitumens," by H. A. Halleck (1841), pp. 36, 39, 144 and 145; **Can. Pat.** 40057 of Aug. 26, 1892 to W. L. Jamieson and F. C. Jamieson.

287 [p. 641] **U. S. Pats.** 3598 of May 25, 1844 to Edouard Deutsch; 14267 of Feb. 12, 1856 to C. C. Hoff; 17851 of Jul. 21, 1857 to J. B. Wands; 21246 of Aug. 24, 1858 to Abram Davis; 26868 of Jan. 17, 1860 to Hiram Tucker; 29423 of Jul. 31, 1860 to J. B. Wands; 32111 of Apr. 23, 1861 to D. S. Anderson; 35526 of Jun. 10, 1862 to J. W. Kingman; 36484 of Sep. 16, 1862 to Alfred Robinson; 38646 of May 26, 1863 to D. M. Ayer; 48311 of Jun. 20, 1865 to Alfred Robinson; 50177 of Sep. 22, 1865 to Robert Skinner, George Duncan and Cesare Merighi; Reissue 2741 of Aug. 20, 1867 to Alfred Robinson; 71210 of Nov. 19, 1867 to Antonio Pelletier; 80207 of Jul. 21, 1868 to Alfred Paraf; 89395 of Apr. 27, 1869 to B. F. Field; 90984 of May 18, 1869 to J. E. Dotch and Edward Duempelman; 97169 of Nov. 23, 1869 to B. R. Croasdale; 103536 of May 31, 1870 to T. R. Abbott; 119059 of Sep. 19, 1871 to H. N. Stimson; 128599 of Jul. 2, 1872 to W. B. Davies; 151683 of Jun. 9, 1874 to Elias Burnham; 187748 of Feb. 27, 1877 to J. C. Cheatham; 204829 of Jun. 11, 1878 to W. P. Jenney; 254461 of Mar. 7, 1882 to J. D. Cheever; 263668 of Aug. 29, 1882 to W. H. Stelwagon; 275841 of Apr. 17, 1883 to H. M. Miner; 285490 of Sep. 25, 1883 to Josiah Jowitt; 304457 of Sep. 2, 1884 to L. L. Sagendorph; 314429 of Mar. 24, 1885 to W. H. H. Childs; 351557 of Oct. 26, 1886 to R. T. Wray and James Thomson; 358502 of Mar. 1, 1887 to G. W. Swan; 366857 of Jul. 19, 1887 to G. W. McGraw; 380065 of Mar. 27, 1888 to B. C. Waite; 380915 of Apr. 10, 1888 to H. M. Miner; 385057 of Jun. 26, 1888 to Alexander Jones; 422609 of Mar. 4, 1890 to J. A. McGraw, G. W. McGraw, Jr., H. L. McGraw and C. E. McGraw; 455000 of Jun. 30, 1891 to M. C. Kerbaugh; 794206 of Jul. 11, 1905 to R. R. Walker; 1231687 of Jul. 3, 1917 to W. H. Adams; 1347095 of Jul 20, 1920 to A. B. Harrisen; 1577890 of Mar. 23, 1926 to C. D. Abraham; **Can. Pats.** 13101 of Jul. 16, 1881 to J. W. Peterson; 15372 of Aug. 26, 1882 to W. O. Callender; 50557 of Nov. 13, 1895 to E. F. Badgley and A. P. Phillips; **Brit. Pats.** of 1763 (Aug. 1), 787 to Nathan Smith; of 1855 (Aug. 27), 1937 to E. C. F. Sautelet; of 1856 (May 26), 1257 to F. C. Jeune; of 1856 (Jul. 24), 1756 to G. T. Bousfield; of 1861 (Feb. 1), 275 to J. J. de Arrieta; of 1868 (Oct. 24), 3249 to R. Ferguson and G. Lord; of 1869 (Jan. 29), 275 to N. C. Szerelmey; of 1871 (Apr. 19), 1035 to W. E. Newton; of 1876 (May 31), 2295 to J. S. Norrie; of 1876 (Jun. 16), 2496 to J. G. Zoller and D. Scott; of 1877 (Mar. 16), 1060 to R. Taylor; of 1878 (Aug. 30), 3436 to H. H. Henson; of 1881 (Jun. 27), 2815 to A. M. Clark; of 1881 (Oct. 11), 4408 to W. O. Callender; of 1885 (Oct. 31), 13140 to J. E. A. Pierret; of 1888 (Mar. 5), 3354 to Thomas Thomson; of 1888 (Apr. 14), 5577 to W. P. Thompson; of 1889 (May 27), 8705 to A. N. Ford; of 1891 (Feb. 17), 2865 to H. F. Williams; of 1891 (Mar. 17), 4750 to Soc. anon. des Ardoisières de Deville and V. van der Heyden; of 1893 (Sep. 9), 17003 to Emille Pierret; of 1897 (Apr. 1), 8343 to J. D. Blackwell; of 1898 (Oct. 21), 22141 to Thomas Thomson; of 1899 (Apr. 6), 7237 to A. Camille; of 1899 (Jul. 22), 15125 to J. Erskine and T. M. Erskine; of 1900 (Apr. 23), 7503 to T. K. Muir; of 1902 (May 14), 11035 to W. Torkington; of 1902 (Nov. 12), 24864 to W. W. Pilkington and W. R. Ormandy; of 1907 (Mar. 27), 7373 to C. A. Peck; **Ger. Pats.** 3097 of Mar. 7, 1878 to F. A. Malchow; 14989 of Jan. 5, 1881 to Adolph Ismer; 85563 of Mar. 14, 1895 to Rudolph Wiggert; 100700 of Nov. 26, 1898 to Carl Baswitz.

288 [p. 641] **U. S. Pats.** 77257 of Apr. 28, 1868 to J. H. Cole; 78014 of May 19, 1868 to Edmond Richardson and J. H. Cole; 83575 of Oct. 27, 1868 to J. J. Wiggin; 84074 of Nov. 17, 1868 to J. W. Wheeler; 91577 of Jun. 22, 1869 to H. G. Soules; 93859 of Aug. 17, 1869 to J. M. Cobb; 95974 of Oct. 19, 1869 to R. O. Benton; 97169 of Nov. 23, 1869 to B. R. Croasdale; 101473 of Apr. 5, 1870 to Samuel Kingan; 104887 of Jun. 28, 1870 to I. L. G. Rice; 113604 of Apr. 11, 1871 to J. J. Wiggin; 114139 of Apr. 25, 1871 to Dwight Hitchcock and Willis Gibbs; 114174 of Apr. 25, 1871 to D. W. McConnell and W. W. Pierce; 120153 of Oct. 24, 1871 to C. L. Fowler; 124794 of Mar. 19, 1872 to D. G. Conger; 125517 of Apr. 9, 1872 to J. H. Barker; 128367 of Jun. 25, 1872 to W. B. Davies; 151683 of Jun. 9, 1874 to Elias Burnham; 179828, 179829 and 179830 of Jul. 11, 1876 to C. M. Warren; 180081 of Jul 18,

1876 to C. M. Warren; 180996 of Aug. 15, 1876 to C. L. Fowler; 191208 of May 22, 1877 to C. M. Warren; 282139 of Jul. 31, 1883 to Welcome White; 284891 of Sep. 11, 1883 to J. F. Perry; 345399 of Jul. 13, 1886 to C. M. Warren; 348996 of Sep. 14, 1886 to T. J. Pearce and M. W. Beardsley; 351557 of Oct. 26, 1886 to R. T. Wray and James Thomson; 358502 of Mar. 1, 1887 to G. W. Swan; 380065 of Mar. 27, 1888 to B. C. Waite; 380915 of Apr. 10, 1888 to H. M. Miner; 437033 of Sep. 23, 1890 to R. S. Merrill; 542701 of Jul. 16, 1895 to E. F. Badgley; 820694 of May 15, 1906 to L. A. Bond; 1155443 of Oct. 5, 1915 to Jean Rosen; 1273673 of Jul. 23, 1918 to Jean Rosen; **Can. Pats.** 6606 of Sep. 28, 1876 to C. M. Warren; 13457 and 13458 of Sep. 20, 1881 to C. M. Warren; 17564 of Sep. 1, 1883 to Welcome White; 97981 of Mar. 13, 1906 to H. A. Parkin; **Brit. Pats.** of 1844 (Mar. 6), 10092 to Thomas Forster; of 1863 (Jan. 27), 232 to H. H. Henson; of 1876 (Dec. 23), 4966 to O. Brooke; of 1879 (Jun. 27), 2596 to W. B. Ritchie; of 1885 (Feb. 5), 1604 to T. J. Pearce and M. W. Beardsley; of 1888 (Feb. 21), 2543 to H. G. Boston and R. Combe; of 1897 (Apr. 1), 8343 to J. D. Blackwell; **Ger. Pat.** 92308 of Apr. 12, 1895 to A. W. Andernach; **Austrian Pat.** 71551 of Apr. 25, 1916 to Schatz & Hürner.

289 [p. 641] "Granularmetric analyses of the talc used as "dusting finish" will show from 5 to 25 per cent of particles passing an 80-, but retained on a 100-mesh sieve, the former applying to finely ground talcs and the latter to coarsely bolted products.

290 [p. 642] "Standard Specs. for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs" (D 250–27), A.S.T.M. Standards 1936, II, 1120.

291 [p. 642] "Standard Specs. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs" (D 226–27), A.S.T.M. Standards 1936, II, 1137.

292 [p. 642] "Federal Spec. for Felt; Asphalt-Saturated (for) Flashings, Roofing, and Waterproofing" HH-F-191, Federal Standard Stock Catalogue, Section IV, (Part 5), Aug. 1, 1933.

293 [p. 642] "Standard Specs. for Coal-Tar-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs" (D 227-27), A.S.T.M. Standards 1936, II, 1140.

294 [p. 642] "Federal Spec. for Felt; Coal-Tar-Saturated (for) Roofing and Waterproofing" HH-F-201, Federal Standard Stock Catalogue, Section IV, (Part 5), Jun. 6, 1933.

295 [p. 642] "Standard Specs. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing" (D 173–27), A.S.T.M. Standards 1936, II, 1134.

296 [p. 642] "Federal Spec. for Cotton-Fabric; Woven, Asphalt-Saturated" HH-C-581a, Federal Standard Stock Catalogue, Section IV, (Part 5), Dec. 18, 1935; "Federal Spec. for Cotton-Fabric; Woven, Coal-Tar-Saturated" HH-C-591, Federal Standard Stock Catalogue, Section IV, (Part 5), Jul. 3, 1935.

297 [p. 642] "Standard Specs. for Burlap Saturated with Bituminous Substances for Use in Waterproofing" (D 174-25), A.S.T.M. Standards 1936, II, 1131.

298 [p. 644] **U. S. Pat.** 1481430 of Jan. 22, 1924 to F. C. Overbury.

299 [p. 645] **U. S. Pats.** 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1536479 of May 5, 1925 to H. A. Cumfer; 1552421 of Sep. 8, 1925 to H. A. Cumfer; 1610749 of Dec. 14, 1926 to H. A. Cumfer; 1701878 of Feb. 12, 1929 to William Freegard; 1752751 of Apr. 1, 1930 to A. E. F. Moone; **Can. Pats.** 224111 of Sep. 26, 1922 to Guyton & Cumfer Mfg. Co.; 244601 and 244602 of Nov. 18, 1924 to Guyton & Cumfer Mfg. Co.

300 [p. 645] **U. S. Pats.** 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1652518 of Dec. 13, 1927 to H. A. Cumfer.

301 [p. 645] **U. S. Pat.** 1536479 of May 5, 1925 to H. A. Cumfer.

302 [p. 645] **U. S. Pat.** 1508959 of Sep. 16, 1924 to H. A. Cumfer; 1511187 of Oct. 7, 1924 to George Ritter; 1610749 of Dec. 14, 1926 to H. A. Cumfer.

303 [p. 645] **U. S. Pats.** 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1830741 of Nov. 3, 1931 to David Low; 1990406 of Feb. 5, 1935 to A. L. Hurxthal and E. B. Kerst; 2043795 of Jun. 9, 1936 to C. A. Dickhaut and C. C. Willis; **Can. Pats.** 219426 of Jun. 6, 1922 to Guyton & Cumfer Mfg. Co.; 304993 of Oct. 21, 1930 to Patent & Licensing Corp.

304 [p. 645] **U. S. Pats.** 1400310 of Dec. 13, 1921 to H. M. Nicholls; 1525829 of Feb. 10, 1925 to Walter Rogers; 1871013 of Aug. 9, 1932 to W. M. Shakespeare.

305 [p. 645] **U. S. Pats.** 1485340 of Feb. 26, 1924 to George Ritter; 1520014 of Dec. 23, 1924 to H. A. Cumfer; 1574835 of Mar. 2, 1926 to Robert Maclean; 1995032 of Mar. 19, 1935 to A. G. Leonard, Jr.; **Can. Pat.** 260251 of Apr. 27, 1906 to Guyton & Cumfer Mfg. Co.; **Ger. Pats.** 207814 of Nov. 24, 1907 to Hermann Paul and Emil Ziaja; 239242 of Jun. 20, 1909 to Hermann Paul; 305489 of Jul. 18, 1917 to Maschinenfabrik Calbe a.S., G.m.b.H.; 550664 of Dec. 9, 1930 to Gose & Werner Maschinenfabrik.

306 [p. 645] See Chapter 24, Reference 2.

307 [p. 645] **U. S. Pat.** 1536479 of May 5, 1928 to H. A. Cumfer.

308 [p. 645] **U. S. Pats.** 1574889 of Mar. 2, 1926 to W. T. Hofmann; 1701878 of Feb. 12, 1929 to William Freegard; 1876761 of Sep. 13, 1932 to L. S. Rosener; 1931430 of Oct. 17, 1933 to P. W. Bur; 1931439 of Oct. 17, 1933 to A. O. Hurxthal; **Ger. Pat.** Design 1319360 of Oct. 17, 1934 to Otto Kinne.

309 [p. 645] **U. S. Pats.** 1567919 of Dec. 29, 1925 to H. A. Cumfer; 1752751 of Apr. 1, 1930 to A. E. F. Moone; 1786992 of Dec. 30, 1930 to F. G. Gronemeyer; 1862256 of Jun. 7, 1932 to H. A. Cumfer; **Can. Pat.** 317445 of Nov. 24, 1931 to Lehon Co.; **Ger. Pat.** 390029 of Feb. 24, 1922 to Johann Gmeindl; Design 1366142 of Feb. 7, 1936 to P. F. Freund & Co.; Design 1376878 of May 16, 1936 to P. F. Freund & Co.

310 [p. 645] **U. S. Pats.** 1467841 of Sep. 11, 1923 to H. A. Cumfer; 1601784 of Oct. 5, 1926 to H. H. Wanders; 1861374 of May 31, 1932 to H. R. Wood; 1872018 of Aug. 16, 1932 to E. T. Street; 1892670 of Jan. 3, 1933 to Fred Jaeger; **Can. Pats.** 232960 of Jul. 24, 1923 to Guyton & Cumfer Mfg. Co.; 263794 of Aug. 24, 1926 to H. H. Wanders.

311 [p. 645] **U. S. Pats.** 1107762 of Aug. 18, 1914 to H. A. Cumfer; 1187716 of Jun. 20, 1916 to H. A. Cumfer; 1257321 of Feb. 26, 1918 to H. A. Cumfer and O. D. McFarland; 1274388 of Aug. 6, 1918 to C. A. Cooney; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1313224 of Aug. 12, 1919 to Gottfrid Hultberg; 1365741 of Jan. 18, 1921 to A. S. Speer; 1435298 of Nov. 14, 1922 to J. L. Hildebrand and H. H. Wanders; 1435353 of Nov. 14, 1922 to H. H. Wanders; 1464309 of Aug. 7, 1923 to H. A. Cumfer; 1467841 of Sep. 11, 1923 to H. A. Cumfer; 1481183 of Jan. 15, 1924 to F. A. Browne; 1492903 of May 6, 1924 to E. T. Street; 1501162 of Jul. 15, 1924 to H. A. Cumfer; 1535678 of Apr. 28, 1925 to J. E. Miller; 1548107 of Aug. 4, 1925 to E. T. Street; 1552447 of Sep. 8, 1924 to Hugo Reichel; 1567920 of Dec. 29, 1925 to H. A. Cumfer; 1569032 and 1569033 of Jan. 12, 1926 to Hugo Reichel; 1600958 of Sep. 21, 1926 to W. J. Hart and R. T. Boas; 1676911 of Jul. 10, 1928 to C. J. McDavitt; 1687823 of Oct. 16, 1928 to O. D. McFarland; 1701760 of Feb. 12, 1929 to P. J. Paffen; 1744650 of Jan. 21, 1930 to E. A. Leonard; 1751562 and 1751563 of Mar. 25, 1930 to Daniel Stinger; 1780716 of Nov. 4, 1930 to E. C. Otis; 1786992 of Dec. 30, 1930 to F. G. Gronemeyer; 1841017 and 1841018 of Jan. 12, 1932 to T. H. Eickhoff; 1847767 of Mar. 1, 1932 to H. C. Koch; 1870443 of Aug. 9, 1932 to D. A. Cumfer; 1871707 of Aug. 16, 1932 to E. E. Klumpmeyer and A. A. Maxwell; 1892680 of Jan. 3, 1933 to E. S. Petersen; 1894037 of Jan. 10, 1933 to N. P. Harshberger; 1915376 of Jun. 27, 1933 to A. E. F. Moone; 1953680 of Apr. 3, 1934 to J. V. Johansen; 1986139 of Jan. 1, 1935 to D. A. Cumfer; 2010887 of Aug. 13, 1935 to A. S. Peterson; Reissue 19689 of Sep. 3, 1935 to N. P. Harshberger; 2020027 of Nov. 5, 1935 to J. L. Gore; 2055738 of Sep. 29, 1936 to E. H. Venrick; **Can. Pats.** 214179 of Nov. 8, 1921 to Roofing Patents Co.; 216870 of Mar. 14, 1922 to Roofing Patents Co.; 231876 of Jun. 12, 1923 to J. L. Hildebrand and H. H. Wanders; 231877 of Jun. 12, 1933 to H. H. Wanders; 312185 of Jun. 9, 1931 to Patent & Licensing Corp.; 312825 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co.

312 [p. 646] "Tentative Specs. for Asphalt Roofing Surfaced with Powdered Talc or Mica" (D 224–35 T), A.S.T.M. Tentative Standards 1936, 827.

313 [p. 646] "Federal Spec. for Roofing; Asphalt-Prepared, Smooth-Surfaced" SS-R-501, Federal Standard Stock Catalogue, Section IV, (Part 5), Aug. 1, 1933.

314 [p. 646] "Underwriters' Labratories Standards for Class C Sheet-Roofing and Shingles Composed of Rag-felt Saturated with Asphalt and Surfaced with Mineral-Surfacing Materials," issued by the Underwriters' Laboratories Inc., Chicago, Ill. (established and maintained by the National Board of Fire Underwriters), Edition of Feb. 23, 1934 (Revised Jun. 19, 1935).

315 [p. 646] "Tentative Specs. for Asphalt Roofing Surfaced with Coarse Mineral Granules" (D 249-35T), A.S.T.M. Tentative Standards 1936, 819.

316 [p. 646] "Tentative Specs. for Asphalt Roofing Surfaced with Fine Mineral Granules" (D 248-35T), A.S.T.M. Tentative Standards 1936, 823.

317 [p. 646] "Federal Spec. for Roofing and Shingles; Asphalt-Prepared, Mineral-Surfaced" SS-R-521, Federal Standard Stock Catalogue, Section IV, (Part 5), Aug. 1, 1933.

318 [p. 646] "Tentative Specs. for Asphalt Cap-Sheet Surfaced with Mineral Granules" (D 371-33T), A.S.T.M. Tentative Standards 1936, 809.

319 [p. 646] "Federal Spec. for Roofing; Asphalt and Asbestos, Mineral-Surfaced" SS-R-511, Federal Standard Stock Catalogue, Section IV, (Part 5), Jul. 9, 1926.

320 [p. 646] "Tentative Specs. for Asphalt Shingles Surfaced with Coarse Mineral Granules" (D 225-35T), A.S.T.M. Tentative Standards 1936, 831.

321 [p. 648] **U. S. Pat.** 1756947 of May 6, 1930 to R. T. Johnston; **Can. Pat.** 247138 of Feb. 24, 1925 to R. T. Johnston; **Ger. Pat.** 322986 of Aug. 8, 1916 to W. Kührmeyer-Franke & Klauer, G.m.b.H.

322 [p. 648] **U. S. Pats.** 876008 and 876010 of Jan. 7, 1908 to F. C. Overbury; 1765834 of Jun. 24, 1930 to G. P. Heppes; **Brit. Pat.** of 1907 (Dec. 24), 28405 to H. W. Lake.

323 [p. 648] **U. S. Pats.** 1286077 of Nov. 26, 1918 to F. C. Overbury; 1765778 of Jun. 24, 1930 to A. E. Schutte; **Can. Pat.** 188744 of Feb. 18, 1919 to F. C. Overbury (Flintkote Co.)

324 [p. 649] **U. S. Pat.** 1315362 of Sep. 9, 1919 to H. A. Cumfer.

325 [p. 649] **U. S. Pat.** 1392498 of Oct. 4, 1921 to O. A. Heppes and C. E. Rahr.

326 [p. 649] **U. S. Pat.** 1668563 of May 8, 1928 to R. T. Johnston; **Can. Pat.** 259400 of Mar. 30, 1926 to R. T. Johnston.

327 [p. 649] **U. S. Pats.** 1400310 of Dec. 13, 1921 to H. M. Nicholls; 1443971 of Feb. 6, 1923 to Alfred Anderson; 1443975 of Feb. 6, 1923 to T. F. Caldwell; 1603976 of Oct. 19, 1926 to H. M. Nicholls; 1845317 of Feb. 16, 1932 to A. E. F. Moone; **Can. Pats.** 220683 of Jul. 11, 1922 to H. M. Nicholls; 297091 of Jan. 28, 1930 to Guyton & Cumfer Mfg. Co.

328 [p. 650] **U. S. Pats.** 1289507 of Dec. 31, 1918 to L. S. Mason; 1290959 of Jan. 14, 1919 to R. L. Fowler.

329 [p. 650] **U. S. Pat.** 1182414 of May 9, 1916 to F. C. Overbury.

330 [p. 650] **U. S. Pat.** 1892672 of Jan. 3, 1933 to W. S. Johnston.

331 [p. 650] **U. S. Pat.** 1574890 of Mar. 2, 1926 to W. T. Hofmann; **Can. Pat.** 229987 of Apr. 3, 1923 to W. T. Hofmann.

332 [p. 650] **U. S. Pat.** 1357920 of Nov. 2, 1920 to Herbert Abraham.

333 [p. 650] **U. S. Pats.** 1131595 of Mar. 9, 1915 to C. S. Bird and G. R. Wyman; 1187259 of Jun. 13, 1916 to B. G. Casler.

334 [p. 650] **U. S. Pats.** 1366146 of Jan. 18, 1921 to J. C. Woodley; 1384110 of Jul. 12, 1921 to J. C. Woodley; 1409767 and 1409768 of Mar. 14, 1922 to R. P. Perry.

335 [p. 650] **U. S. Pat.** 427146 of May 6, 1890 to Hermann Bormann; **Can. Pat.** 364018 of Feb. 9, 1937 to J. A. Topping.

336 [p. 650] **Brit. Pat.** 450412 of Jan. 16, 1935 to D. L. Irwin.

337 [p. 650] **U. S. Pats.** 490668 of Jan. 31, 1893 to G. S. Lee; 624976 of May 16, 1899 to R. J. Redick; 753982 of Mar. 8, 1904 to S. R. Holland; 1544144 of Jun. 30, 1925 to Dozier Finley.

338 [p. 650] **U. S. Pats.** 1592760 of Jul. 13, 1926 to A. C. Fischer; 1759999 of May 27, 1930 to C. E. Rahr; 1769628 of Jul. 1, 1930 to A. C. Fischer.

339 [p. 650] **U. S. Pat.** 767723 of Aug. 16, 1904 to F. W. Terpening; 1595079 of Aug. 10, 1926 to A. C. Fischer.

340 [p. 650] **U. S. Pat.** 1618192 of Feb. 22, 1927 to G. P. Heppes; **Can. Pat.** 249095 of Apr. 28, 1925 to G. P. Heppes.

341 [p. 650] **U. S. Pats.** 1586892 of Jun. 1, 1926 to A. C. Fischer; 1690792 of Nov. 6, 1928 to R. C. Neptune; 1710104 of Apr. 23, 1929 to R. C. Neptune.

342 [p. 650] **Can. Pat.** 221937 of Aug. 8, 1922 to J. T. Sullivan.

343 [p. 650] **U. S. Pat.** 1741403 of Dec. 31, 1929 to M. L. Caton.

344 [p. 650] **U. S. Pats.** 1715080 of May 28, 1929 to O. A. Frederickson; 1769627 of Jul. 1, 1930 to A. C. Fischer.

345 [p. 650] **U. S. Pats.** 1327933 of Jan. 13, 1920 to J. H. Young; 1410790 of Mar. 28, 1922 to J. H. Young; 1536549 of May 5, 1925 to J. H. Young; 1539512 of May 26, 1925 to Thomas Robinson; 1597168 of Aug. 24, 1926 to D. M. Sutherland, Jr.; 1640795 of Aug. 30, 1927 to J. S. Miller, Jr.; 1672579 of Jun. 5, 1928 to C. H. Rayner; 1715080 of May 28, 1929 to O. A. Frederickson; 1871982 of Aug. 16, 1932 to H. D. Geyer; **Can. Pats.** 244618 of Nov. 18, 1924 to H. H. Robertson Co.; 282856 of Aug. 28, 1928 to Barber Asphalt Co.; **Brit. Pats.** 132522 of Sep. 11, 1918 to H. H. Robertson Co.; 159863 and 159864 of Jan. 4, 1921 to H. H. Robertson Co.; 209727 of Jan. 11, 1923 to J. H. Young; 243614 of Jan. 20, 1926 to C. M. Taylor.

346 [p. 650] **U. S. Pat.** 314429 of Mar. 24, 1885 to W. H. H. Childs.

347 [p. 650] **U. S. Pat.** 1919677 of Jul. 25, 1933 to J. H. Young.

348 [p. 650] **U. S. Pat.** 1904341 of Apr. 18, 1933 to J. H. Young.

349 [p. 650] **U. S. Pats.** 1800193 and 1800194 of Apr. 7, 1931 to A. C. Fischer.

350 [p. 650] **U. S. Pat.** 1314753 of Sep. 2, 1919 to F. C. Overbury.

351 [p. 650] **U. S. Pat.** 1238805 of Sep. 4, 1917 to F. C. Overbury.

352 [p. 650] **U. S. Pat.** 1289328 of Dec. 31, 1918 to H. R. Wardell.

353 [p. 650] **U. S. Pat.** 1934315 of Nov. 7, 1933 to Morris Levine; **Can. Pat.** 357604 of May 5, 1936 to Carborundum Co.

354 [p. 651] **U. S. Pat.** 1963752 of Jun. 19, 1934 to A. D. MacNutt; **Can. Pat.** 365511 of Apr. 20, 1937 to Certainteed Products Corp.

355 [p. 651] **U. S. Pat.** 2022429 of Nov. 26, 1935 to C. J. Merriam.

356 [p. 651] **U. S. Pat.** 1808590 of Jun. 2, 1931 to A. W. Andernach; **Ger. Pats.** 530529 of Sep. 4, 1928 to G. A. Jahn; 531761 of Jul. 6, 1929 to G. A. Jahn; 543733 of Mar. 28, 1931 to G. A. Jahn; Design 1213324 of Mar. 15, 1932 to A. W. Andernach; Design 1228855 of Jun. 9, 1932 to A. W. Andernach.

357 [p. 651] **Ger. Pat.** 448826 of Aug. 11, 1927 to Ludwig Schwabe.

358 [p. 652] **U. S. Pats.** 1447265 of Mar. 6, 1923 to A. R. Purdy; **Can. Pat.** 230271 of Apr. 10, 1923 to Ruberoid Co., Ltd.; **Brit. Pat.** 179130 of Aug. 16, 1921 to Ruberoid Co.

359 [p. 652] **U. S. Pats.** 1480023, 1480024 and 1480025 of Jan. 8, 1924 to A. S. Speer; 1640678 and 1640679 of Aug. 30, 1927 to A. S. Speer; **Can. Pat.** 218271 of May 2, 1922 to Roofing Patents Co.

360 [p. 652] **Ger. Pats.** 573115 of Aug. 11, 1928 to Alexander Malchow and Wolfgang Malchow; Design 1251409 of Oct. 29, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.; Design 1253500 of Nov. 7, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.

361 [p. 652] **Ger. Pats.** 344351 of Apr. 12, 1921 to Kurt Haurwitz; Design 1234390 of Aug. 29, 1932 to Papierfabrik Krampe, A.-G.

362 [p. 652] **Ger. Pat.** 600565 of Nov. 24, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.

363 [p. 652] **Ger. Pat.** Design 1253499 of Nov. 7, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.

364 [p. 653] **U. S. Pat.** 820470 of May 15, 1906 to R. W. Bird.

365 [p. 653] **U. S. Pat.**, 1833471 of Nov. 24, 1931 to A. N. Parrett; **Ger. Pats.** 402251 of Nov. 7, 1922 to Chemisches Laboratorium für Anstrichstoffe G.m.b.H. 414483 of May 24, 1924 to Chemisches Laboratorium für Anstrichstoffe, G.m.b.H.; 477613 of Mar. 20, 1924 to Forschungs- und Lehrinstitut für Anstreichtechnik, G.m.b.H.

366 [p. 653] **U. S. Pats,** 23882 of May 3, 1859 to N. A. Dyar; 43171 of Jun. 21, 1864 to S. M. Allen; 44220 of Sep. 24, 1864 to Alfred Robinson; 48311 of Jun. 20, 1865 to Alfred Robinson; Reissue 2741 of Aug. 20, 1867 to Alfred Robinson; 70028, 70029 and 70030 of Oct. 22, 1867 to John Scanlan; 75197 of Mar. 3, 1868 to Alfred Robinson; 104380 of Jun. 14, 1870 to Edward Van Orden; Reissue 4862 of Apr. 9, 1872 to Alfred Robinson; 134002 of Dec. 17, 1874 to G. W. Pond; 202902 of Apr. 23, 1878 to C. M. Warren; 209830 of Nov. 12, 1878 to Tobias New; 211669 of Jan. 28, 1879 to W. H. Rankin; 226461 of Apr. 13, 1880 to W. H.

Stewart; 237158 of Feb. 1, 1881 to R. A. Bendall; 251921 of Jan. 3, 1882 to Tobias New; 256368 of Apr. 11, 1882 to G. H. Pöschel; 269786 of Dec. 26, 1882 to S. H. Hamilton; 278278 of May 22, 1883 to Augustine Sackett; 278722 of Jun. 5, 1883 to H. M. Miner; 291440 of Jan. 1, 1884 to C. M. Warren; 291600 of Jan. 8, 1884 to Josiah Jowitt; 291628 of Jan. 8, 1884 to Augustine Sackett; 293491 and 293492 of Feb. 12, 1884 to H. M. Miner; 302938 of Aug. 5, 1884 to W. H. Rankin; 312451 of Feb. 17, 1885 to Michael Ehret, Jr.; 318910 of May 26, 1885 to Josiah Jowitt; 332570 of Dec. 15, 1885 to W. H. Stewart; 341043 of May 4, 1886 to Tobias New; 351948 of Nov. 2, 1886 to C. M. Warren; 352619 of Nov. 16, 1886 to F. L. Kane; 354311 of Dec. 14, 1886 to C. A. Favel; 361050 of Apr. 12, 1887 to W. H. H. Childs; 362202 of May 3, 1887 to Phillip Carey; 366857 of Jul. 19, 1887 to G. W. McGraw; 372894 of Nov. 8, 1887 to W. H. H. Childs; 373085 of Nov. 15, 1887 to H. M. Miner; 385057 of Jun. 26, 1888 to Alexander Jones; Reissue 11017 of Jul. 30, 1889 to J. A. McGraw, H. L. McGraw, G. W. McGraw, Jr. and C. E. McGraw; 418569 of Dec. 31, 1889 to H. W. Johns; 427124 of May 6, 1890 to M. C. Kerbaugh; 427147 of May 6, 1890 to Herman Bormann; 429885 of Jun. 10, 1890 to W. H. H. Childs; 453979 of Jun. 9, 1891 to G. S. Lee; 455000 of Jun. 30, 1891 to M. C. Kerbaugh; 542701 of Jul. 16, 1895 to E. F. Badgley; 565336 of Aug. 4, 1896 to Michael Ehret, Jr.; 674219 of May 14, 1901 to J. A. Scharwath; 800320 of Sep. 26, 1905 to T. F. Odell; 845290 of Feb. 26, 1907 to E. H. Binns; 851331 of Apr. 23, 1907 to H. R. Wardell; 1083243 of Dec. 30, 1913 to W. C. Edwards, Jr.; 1154875 of Sep. 28, 1915 to James Meade; 1320549 of Nov. 4, 1919 to O. A. Heppes; 1483711 of Feb. 12, 1924 to Maurice Blumenthal; 1606428 of Nov. 9, 1926 to Lester Kirschbraun; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1644652 of Oct. 4, 1927 to Lester Kirschbraun; 1676351 of Jul 10, 1928 to Thomas Robinson; 1694523 of Dec. 11, 1928 to J. F. White; 1788121 of Jan. 6, 1931 to F. C. Overbury; 1889177 of Nov. 29, 1932 to C. L. Keller; **Can. Pats.** 16821 of May 5, 1883 to G. H. Poschel; 22642 of Oct. 19, 1885 to D. G. Conger; 38156 of Jan. 26, 1892 to H. W. Johns; 64448 of Oct. 18, 1899 to J. W. Paterson; 106875 of Aug. 6, 1907 to Barber Asphalt Paving Co.; **Brit. Pats.** of 1860 (Oct. 31), 2666 to J. Anderson; of 1868 (Apr. 22), 1308 to T. Whittaker; of 1873 (Sep. 26), 3147 to J. A. Turner; of 1874 (Feb. 4), 447 and 449 to John Macintosh; of 1874 (Mar. 4), 770 to F. Wirth; of 1880 (Oct. 9), 4107 to J. C. Mewburn; of 1882 (Jul. 25), 3539 to Josiah Jowitt and G. S. Page; of 1893 (Jul. 28), 14551 to G. McTear; 367843 of Sep. 23, 1930 to F. B. Dehn; **Ger. Pats.** 24612 of Oct. 5, 1882 to David Röhm; 121436 of May 6, 1899 to A. W. Andernach; Appl. W-51.30 of Apr. 30, 1930 to Fabrik Walter-Falckenberg Nachf. (rejected); **Austrian Pat.** 4000 of Apr. 25, 1901 to Wenzel Němeček; **Swiss Pat.** 17864 of Feb. 8, 1899 to J. Gredig.

367 [p. 653] **U. S. Pats.** 81641 of Sep. 1, 1868 to H. W. Johns; 906252 of Dec. 8, 1908 to J. E. Meek; 1296324 of Mar. 4, 1919 to J. A. Scharwath; 1325546 of Dec. 23, 1919 to H. R. Wardell; 1436914 of Nov. 28, 1922 to W. R. Seigle; **Brit. Pat.** 411672 of May 19, 1933 to D. L. Irwin.

368 [p. 653] **U. S. Pats.** 282139 of Jul. 31, 1883 to Welcome White; 333138 of Dec. 29, 1885 to Francis Line; 418519 of Dec. 31, 1889 to H. W. Johns; 690526 of Jan. 7, 1902 to F. S. Miller and W. B. Davenport; 817619 of Apr. 10, 1906 to G. F. Bishopric; 917543 of Apr. 6, 1909 to A. J. Cohen; 1044558 of Nov. 19, 1912 to W. J. Moeller; 1062400 of May 20, 1913 to S. C. Irving; 1218217 of Mar. 6, 1917 to E. J. Schroder; 1220966 of Mar. 27, 1917 to O. R. Emigh; 1325546 of Dec. 23, 1919 to H. R. Wardell; 1548910 and 1548911 of Aug. 11, 1925 to J. C. Sherman; 1616921 of Feb. 8, 1927 to C. E. Rahr and Lester Kirschbraun; **Can. Pats.** 96317 of Nov. 28, 1905 to G. F. Bishopric; 146427 of Mar. 11, 1913 to W. J. Moeller; 211947 of May 24, 1921 to H. H. Robertson Co.; 278682 of Mar. 20, 1928 to Albert Roby; **Brit. Pats.** of 1889 (Dec. 31), 20976 to H. W. Johns; of 1891 (Oct. 14), 2457 to J. N. Hopper; of 1913 (May 20), 11752 to S. C. Irving; 411672 of May 19, 1933 to Ruberoid Co., Ltd.; **Ger. Pat.** 141760 of May 22, 1901 to Maurice Coutellier; **French Pat.** 772978 of Nov. 9, 1934 to Ruberoid Co., Ltd.

369 [p. 653] **U. S. Pats.** 42910 of May 24, 1864 to J. H. Green; 43171 of Jun. 21, 1864 to S. M. Allen; 70028 of Oct. 22, 1867 to John Scanlan; 118025 of Aug. 15, 1871 to William Kinsey; 125573 and 125574 of Apr. 9, 1872 to H. W. Johns; 150636 of May 5, 1874 to J. A. Turner; 278722 of Jun. 5, 1883 to H. M. Miner; 293491 of Feb. 12, 1884 to H. M. Miner;

293492 of Feb. 12, 1884 to H. M. Miner; 304744 of Sep. 9, 1884 to H. M. Miner; 322153 of Jul. 14, 1885 to L. F. Blair and J. W. Roche; 348844 of Sep. 7, 1886 to David Harger; 385057 of Jun. 26, 1888 to Alexander Jones; 453979 of Jun. 9, 1891 to G. S. Lee; 490668 of Jan. 31, 1893 to G. S. Lee; 624976 of May 16, 1899 to R. J. Redick; 636022 of Oct. 31, 1899 to G. D. Crabbs and W. H. Pendery; 753982 of Mar. 8, 1904 to S. R. Holland; 775968 of Nov. 29, 1904 to August Gross; 813336 of Feb. 20, 1906 to H. R. Wardell; 820470 of May 15, 1906 to R. W. Bird; 845414 of Feb. 26, 1907 to Samuel Herbert; 1040706 of Oct. 8, 1912 to L. F. Lindley; 1119697 of Dec. 1, 1914 to G. J. Hoffman; 1152798 of Sep. 7, 1915 to Julius de Long and J. B. d'Homergue; 1226904 of May 22, 1917 to R. C. Neptune; 1248909 of Dec. 4, 1917 to H. B. Pullar; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; 1642316 of Sep. 13, 1927 to H. R. Wardell; 1644050 of Oct. 4, 1927 to W. H. Cady; **Can. Pat.** 2557 of Jul. 25, 1873 to Edward Churchill; **Brit. Pats.** of 1859 (Aug. 24), 1936 to T. Briggs; of 1860 (Jul. 26), 1722 to W. E. Newton; of 1860 (Aug. 14), 1969 to R. D. McKibbin; of 1871 (Mar. 9), 630 to J. A. Turner; of 1871 (Nov. 4), 2971 to T. Briggs; of 1873 (Sep. 26), 3147 to J. A. Turner; of 1875 (Nov. 26), 4109 to P. McLaine; of 1888 (Apr. 14), 5577 to W. P. Thompson; of 1888 (Sep. 28), 13971 to Donald Nicoll; of 1893 (Oct. 14), 17003 to Emille Pierret; **Ger. Pats.** 97894 of Jul. 24, 1897 to J. E. Christensen (Erichsen & Menge); 119360 of Jun. 13, 1900 to Erichsen & Menge; Design 226487 of May 3, 1904 to Hoppe & Roehming; Design 339008 of Apr. 22, 1908 to Fritz Grimm; Design 377108 of Apr. 16, 1909 to Th. Fahnenstich Söhne; Design 1275323 of Aug. 9, 1933 to Emil Meyer; **Swiss Pat.** 35288 of Jan. 15, 1906 to Carl Hohl.

370 [p. 654] **U. S. Pat.** 419120 of Jan. 7, 1890 to David Harger.

371 [p. 654] **U. S. Pats.** 143697 of Oct. 14, 1873 to R. S. Jennings; 391237 of Oct. 16, 1888 to C. M. Garrison; 403588 of May 21, 1889 to G. A. Herdman; 407195 of Jul. 16, 1889 to C. M. Garrison; 418569 of Dec. 31, 1889 to H. W. Johns; 423042 of Mar. 11, 1890 to A. N. Ford; 446775 of Feb. 17, 1891 to J. N. Hopper; 539767 of May 21, 1895 to F. W. Coolbaugh and N. M. Goodlett; 542791 of Jul. 16, 1895 to E. F. Badgley; 767723 of Aug. 16, 1904 to F. W. Terpenning; 1074404 of Sep. 30, 1913 to W. L. Barnhart; 1201756 of Oct. 17, 1916 to R. P. Perry; 1320502 of Nov. 4, 1919 to J. T. Simpson; 1362887 of Dec. 21, 1920 to R. E. Moist; 1498351 of Jun. 17, 1924 to W. L. Clement; 1657979 of Jan. 31, 1928 to F. W. Thomas; 1844655 of Feb. 9, 1932 to Yuichi Hikasa; 2017106 of Oct. 15, 1935 to H. N. Sandell; **Can. Pats.** 22081 of Jul. 14, 1885 to Alfred Ford and J. A. Archer; 35535 of Nov. 29, 1890 to J. N. Hopper; 89633 of Oct. 18, 1904 to F. W. Terpenning; 140265 of May 7, 1912 to A. R. Manson; 220378 of Jul. 4, 1922 to R. E. Moist; **Brit. Pats.** of 1885 (Jan. 16), 626 to E. T. Bellhouse and D. Longsdon; of 1887 (Dec. 19), 17442 to Alfred Ford; of 1897 (Apr. 3), 809 to Eduard Kauert; of 1901 (Jan. 7), 400 to A. Gustorf; of 1901 (Nov. 27), 24139 to J. Scheck; of 1904 (Jul. 8), 15277 to A. Field; 116591 of Jul. 28, 1917 to J. C. Crets; 119494 of Jun. 29, 1917 to E. O. Brown; 120750 of Oct. 19, 1927 to E. O. Brown; **Ger. Pats.** 91809 of May 24, 1896 to Eduard Kauert; 168829 of Jan. 20, 1902 to L. Ebert; 189069 of Sep. 2, 1904 to C. F. Lau; 222959 of Mar. 10, 1909 to Alexander Wendler; 483735 of May 27, 1926 to Otto Engel; 514787 of Oct. 15, 1926 to Otto Engel; 530529 of Sep. 4, 1928 to G. A. Jahn; Appl. M-5.30 of Feb. 8, 1930 to Wilhelm Meusel; Design 430953 of Mar. 3, 1910 to August Hermann; Design 1273729 of Jul. 8, 1933 to Anton Funke; Design 1400264 of Feb. 3, 1937 to Johann Drolshagen; **Austrian Pats.** 202 of Sep. 25, 1899 to Josef Hasenbichler; 49797 of Sep. 11, 1911 to Hermann Schliszke.

372 [p. 654] **U. S. Pats.** 441036 of Nov. 18, 1890 to Arthur Siebel; 788358 of Apr. 25, 1905 to F. D. Jacobs; 816661 of Apr. 3, 1906 to F. D. Jacobs; Reissue 12475 of Apr. 24, 1906 to F. D. Jacobs; 836157 of Nov. 20, 1906 to P. W. Turner; 845290 of Feb. 26, 1907 to E. H. Binns; 1002301 of Sep. 5, 1911 to E. T. Newsome; 1074404 of Sep. 30, 1913 to W. L. Barnhart; 1115714 of Nov. 3, 1914 to T. D. Miller; 1167949 of Jan. 11, 1916 to P. M. Stewart; 1168599 of Jan. 18, 1916 to J. H. Caffrey; Design 49171 of Jun. 13, 1916 to B. S. Annis; 1195090 of Aug. 15, 1916 to H. H. Robertson; 1236994 of Aug. 14, 1917 to P. M. Stewart; 1243808 of Oct. 23, 1917 to J. H. Caffrey; 1277755 of Sep. 3, 1918 to H. H. Robertson; 1277758 of Sep. 3, 1918 to W. W. Roney; 1288734 of Dec. 24, 1918 to P. M. Stewart; 1366999 of Feb. 1, 1921 to J. H. Young; 1418348 and 1418349 of Jun. 6, 1922 to J. H. Young; 1421338

of Jun. 27, 1922 to J. H. Young; 1432512 of Oct. 17, 1922 to J. H. Young; 1529463 of Mar. 10, 1925 to J. R. Burnside; 1611031 of Dec. 14, 1926 to Turner Henderson; 1700561 of Jan. 29, 1929 to F. J. Commin and A. H. J. Wright; 1812732 of Jun. 30, 1931 to J. H. Young; 1863186 of Jun. 14, 1932 to J. E. Burns; 1959610 of May 22, 1934 to J. H. Bowman; 1973004 of Sep. 11, 1934 to C. F. Langworthy and Helen Hughes; 1973103 of Sep. 11, 1934 to J. H. Young and D. S. Hubbell; 2068533 of Jan. 19, 1937 to A. W. Coffman; 2073334 of Mar. 9, 1937 to A. W. Coffman; **Can. Pats.** 35797 of Jan. 16, 1891 to W. W. Green; 96171 of Nov. 21, 1905 to F. D. Jacobs; 105020 of Apr. 30, 1907 to P. W. Turner; 140827 of May 28, 1912 to H. R. Wardell; 230268 of Apr. 10, 1923 to H. H. Robertson Co.; 233564 of Aug. 14, 1923 to H. H. Robertson Co.; 236657 and 236658 of Dec. 25, 1923 to H. H. Robertson Co.; 248597 of Apr. 14, 1925 to J. R. Burnside; 354564 of Dec. 3, 1935 to Ruberoid Co., Ltd.; **Brit. Pats.** of 1855 (Mar. 29), 703 to R. Johnson and W. W. Johnson; of 1891 (Oct. 14), 2457 to J. N. Hopper; of 1895 (Sep. 2), 16399 to Edward Nelson; of 1897 (Apr. 3), 809 to Eduard Kauert; of 1904 (Jun. 2), 12518 to W. L. Watson; of 1912 (Jan. 17), 1360 to H. R. Wardell; 106997 and 106998 of Jun. 3, 1916 to Asbestos Protected Metal Co.; 136984 of Mar. 24, 1919 to T. D. Miller; 156864 of Mar. 9, 1922 to H. H. Robertson Co.; 211746 of Apr. 16, 1923 to George Harrison; 214457 of Apr. 16, 1923 to George Harrison; 333978 of Jun. 6, 1929 to A. H. J. Wright; 355502 of Aug. 14, 1930 to Albert Field; 371683 of Jun. 30, 1931 to J. H. Young; 379188 of Dec. 29, 1931 to J. H. Young; 428916 of Nov. 9, 1934 to G. E. Black; 446978 of Nov. 8, 1934 to E. R. James; **Ger. Pats.** 508656 of Oct. 27, 1926 to Walli Sattig; Design 418900 of Nov. 16, 1909 to Friedrich Bock; Design 421178 of Feb. 8, 1910 to Michael Faist; Design 436615 of Jul. 18, 1910 to Michael Faist; Design 1330980 of Dec. 8, 1934 to Chr. H. Rang.

373 [p. 654] **Ger. Pat.** 126208 of Nov. 17, 1899 to J. Scheck.

374 [p. 654] See Reference 423.

375 [p. 654] **U. S. Pat.** 985140 of Feb. 28, 1911 to Hedley Button.

376 [p. 654] **U. S. Pats.** 1526552 of Feb. 17, 1925 to Lester Kirschbraun; 1526553 of Feb. 17, 1925 to Lester Kirschbraun and T. W. Morse; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; **Can. Pat.** 309604 of Mar. 24, 1931 to J. B. Hamilton.

377 [p. 654] **Ger. Pat.** Design 1247194 of Dec. 17, 1932 to Ludwig Pringal.

378 [p. 655] **U. S. Pats.** 1164965 of Dec. 21, 1915 to H. S. Smalley; 1705015 of Mar. 12, 1929 to A. H. Irving; 1974314 of Sep. 18, 1934 to E. R. Schaeffer; 1984551 of Dec. 18, 1934 to E. R. Schaeffer.

379 [p. 655] **U. S. Pats.** 60381 of Dec. 11, 1866 to M. A. Johnson; 1543131 of Jun. 23, 1925 to J. A. Stough; 1642316 of Sep. 13, 1927 to H. R. Wardell; 1654846 of Jan. 3, 1928 to J. A. Stough.

380 [p. 655] **U. S. Pats.** 238991 of Mar. 15, 1881 to W. H. R. Toye; 249342 of Nov. 8, 1881 to William Hadden; 250301 of Nov. 29, 1881 to W. H. R. Toye; 342315 of May 25, 1886 to Frederick Beck; 358647 of Mar. 1, 1887 to William Campbell; 527416 of Oct. 16, 1894 to Antonio Federici; 674125 of May 14, 1901 to Phillip Semmer; 1082364 of Dec. 23, 1913 to A. S. Spiegel; 1134086 of Mar. 30, 1915 to F. C. Lowrey; 1154334 of Sep. 21, 1915 to F. C. Overbury; 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; Design 48172 of Nov. 23, 1915 to W. P. Dun Lany; 1192601 of Jul. 25, 1916 to D. E. Boismenue and N. J. Wagner; 1194890 of Aug. 15, 1916 to A. S. Spiegel; 1214658 of Feb. 6, 1917 to W. P. Dun Lany; 1236462 of Aug. 14, 1917 to W. F. McKay; 1254481 of Jan. 22, 1918 to C. M. Clarke; 1264343 of Apr. 30, 1918 to A. S. Spiegel; 1264831 of Apr. 30, 1918 to W. F. McKay; 1265315 of May 7, 1918 to S. M. Ford; 1268105 of Jun. 4, 1918 to S. M. Ford; 1269906 of Jun. 18, 1918 to C. M. Clarke; 1290959 of Jan. 14, 1919 to R. L. Fowler; 1294252 of Feb. 11, 1919 to S. M. Ford; 1295360 of Feb. 25, 1919 to F. C. Overbury; 1300591 of Apr. 15, 1919 to Michael Ebinger; 1339327 of May 4, 1920 to S. M. Ford; 1365970 of Jan. 18, 1921 to M. H. Elvidge; 1376092 of Apr. 26, 1921 to O. A. Heppes; 1379368 of May 24, 1921 to A. S. Speer; 1384534 of Jul. 12, 1921 to L. F. Lindley and A. S. Speer: 1394149 of Oct. 18, 1921 to Harry Cumfer and O. D. McFarland; 1398272 of Nov. 29, 1921 to F. C. Overbury; 1414778 of May 2, 1922 to M. H. Elvidge; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1419169 of Jun. 13, 1922 to F. C. Overbury; 1434332 of Oct. 31, 1922 to M. H. Elvidge; 1448203 of Mar. 13,

1923 to Harry Cumfer and O. D. McFarland; 1452978 of Apr. 24, 1923 to J. S. Miller, Jr.; 1456224 of May 22, 1923 to A. E. Currier; 1484760 of Feb. 26, 1924 to H. A. Cumfer; 1513969 of Nov. 4, 1924 to Harry Cumfer and O. D. McFarland; 1517826 of Dec. 2, 1924 to H. A. Cumfer and O. D. McFarland; 1584557 of May 11, 1926 to H. C. Koch; 1609921 of Dec. 7, 1926 to S. G. Wright; 1623189 of Apr. 5, 1927 to Lester Kirschbraun; 1666429 of Apr. 17, 1928 to Charles Stolp, Jr.; 1669166 of May 8, 1928 to C. L. Keller; 1692210 of Nov. 20, 1928 to W. T. Hofmann; 1716072 of Jun. 4, 1929 to A. A. MacCubbin; 1774988 of Sep. 2, 1930 to Robert Maclean; 1791560 of Feb. 10, 1931 to G. P. Heppes; 1791571 of Feb. 10, 1931 to F. C. Overbury; 1794719 of Mar. 3, 1931 to Robert Maclean; 1820005 of Aug. 18, 1931 to Robert Maclean; 1833651 of Nov. 24, 1931 to E. R. Low; 1834917 of Dec. 1, 1931 to F. H. Gilchrist; 1834998 of Dec. 8, 1931 to M. R. Becker; 1857463 of May 10, 1932 to Robert Maclean; 1872622 of Aug. 16, 1932 to E. S. Donahue; 1872628 of Aug. 16, 1932 to J. F. Esch; 1886969 and 1886970 of Nov. 8, 1932 to A. E. F. Moone; 1898989 and 1898990 of Feb. 21, 1933 to N. P. Harshberger; 1900597 of Mar. 7, 1933 to E. C. Otis; 1900598 of Mar. 7, 1933 to E. T. Doyon; 1906471 of May 2, 1933 to B. W. Lambacher; 1915905 of Jun. 27, 1933 to A. S. Speer; 1916095 and 1916096 of Jun. 27, 1933 to D. A. Cumfer; 1927820 of Sep. 26, 1933 to C. R. Eckert; 1928274 and 1928275 of Sep. 26, 1933 to J. L. Wettlaufer; 1933878 of Nov. 7, 1933 to J. D. Stebbins; 1967419 Jul. 24, 1934 to A. E. F. Moone; 1998078 and 1998079 of Apr. 16, 1935 to William Freegard; 2000077 of May 7, 1935 to N. P. Harshberger; 2011006 of Aug. 13, 1935 to Robert Maclean; 2037788 of Apr. 21, 1936 to Gottfried Hultberg; 2037822 of Apr. 21, 1936 to John Robert; 2068761 of Jan. 26, 1937 to B. S. Penley; 2068767 of Jan. 26, 1937 to John Robert; 2074130, of Mar. 16, 1937 to B. S. Penley; 2074131 of Mar. 16, 1937 to B. S. Penley and R. A. Holdsworth; 2074147 of Mar. 16, 1937 to R. A. Holdsworth; 2074445 of Mar. 23, 1937 to J. L. Wettlaufer; **Can. Pats.** 161694 of Mar. 30, 1915 to F. C. Lowrey; 210448, 210452 and 210453 of Apr. 12, 1921 to Roofing Patents Co.; 217362 of Mar. 28, 1922 to Roofing Patents Co.; 251356 of Jul. 7, 1925 to H. A. Cumfer; 255416 of Nov. 17, 1925 to H. A. Cumfer and O. D. McFarland; 260002 of Apr. 20, 1926 to Flintkote Co.; 265816 of Nov. 16, 1926 to Robert Maclean; 266193 of Nov. 30, 1926 to H. A. Cumfer and O. D. McFarland; 266656 of Dec. 14, 1926 to H. C. Koch; 270344 of May 3, 1927 to Flintkote Co.; 270843 of May 24, 1927 to Flintkote Co.; 2801115 of May 15, 1928 to Robert Maclean; 286917 of Feb. 5, 1929 to D. A. Cumfer; 299769 of Apr. 29, 1930 to Patent & Licensing Corp.; 310697 of Apr. 21, 1931 to Patent & Licensing Corp.; 311966 of Jun. 2, 1931 to Patent & Licensing Corp.; 350847 of Jun. 11, 1935 to Barrett Co.; 356757, 356758, 356759 of Mar. 24, 1936 to Barrett Co.; 357588, 357589 and 357590 of May 5, 1936 to Barrett Co.; **Ger. Pats.** Design 1259570 of Mar. 3, 1933 to Dessauer Dachpappen- und Teerprodukte-Fabrik, m.b.H., Mathis & Dr. Wendschuh; Design 1282963 of Nov. 8, 1933 to Alfred Elben; Design 1300519 of Dec. 12, 1933 to Fritz Werner; Design 1303247 of Nov. 11, 1933 to Fritz Werner.

381 [p. 655] **U. S. Pats.** 1214658 and 1214659 of Feb. 6, 1917 to W. P. Dun Lany; 1263051 of Apr. 16, 1918 to S. M. Ford; 1471043 of Oct. 16, 1923 to T. J. Lords and G. P. Lennart; 1471493 of Oct. 23, 1923 to T. J. Lords and G. P. Lennart; 1764080 of Jun. 17, 1930 to R. T. Johnston; **Can. Pats.** 259397 of Mar. 30, 1926 to Flintkote Co.; 266193 of Nov. 30, 1926 to H. A. Cumfer and O. D. McFarland; 356760 and 356761 of Mar. 24, 1936 to Barrett Co.; 356904 of Mar. 31, 1936 to Barrett Co.

382 [p. 655] **U. S. Pats.** Design 45836 of May 26, 1914 to S. H. Goldberg; 1123727 of Jan. 5, 1915 to S. H. Goldberg; 1176049 of Mar. 21, 1916 to S. H. Goldberg; 1236462 of Aug. 14, 1917 to W. F. McKay; 1250577 and 1250578 of Dec. 18, 1917 to S. H. Goldberg; 1264831 of Apr. 30, 1918 to W. F. McKay; 1298690 of Apr. 1, 1919 to S. H. Goldberg; 1345922 of Jul. 6, 1920 to S. H. Goldberg; 1365902 of Jan. 18, 1921 to S. M. Ford; 1499308 of Jun. 24, 1924 to C. E. Rahr; **Can. Pats.** 157859 of Sep. 15, 1914 to S. H. Goldberg; 167268 of Jan. 25, 1916 to S. H. Goldberg; **Brit. Pat.** of 1913 (Apr. 26), 9806 to S. H. Goldberg; **Ger. Pat.** 288749 of Apr. 17, 1914 to S. H. Goldberg.

383 [p. 655] **U. S. Pats.** 1036427 of Aug. 20, 1912 to C. S. Bird; 1080647 of Dec. 9, 1913 to W. F. McKay; 1126114 of Jan. 26, 1915 to A. S. Spiegel; 1208595 of Dec. 12, 1916 to W. F. McKay; 1219652 of Mar. 20, 1917 to W. F. McKay; 1228191 of May 29, 1917 to W. P.

Dun Lany; 1294785 of Feb. 18, 1919 to S. M. Ford; 1296984 of Mar. 11, 1919 to O. D. McFarland; 1351181 of Aug. 31, 1920 to W. F. McKay; 1366146 of Jan. 18, 1921 to J. C. Woodley; 1369129 of Feb. 22, 1921 to Earl Ross; 1384110 of Jul. 12, 1921 to J. C. Woodley; 1409767 of Mar. 14, 1922 to R. P. Perry; 1454323 of May 8, 1923 to Hugh MacInnes; 1469543 of Oct. 2, 1923 to G. L. Strachan and J. I. Strachan; 1472884 of Nov. 6, 1923 to C. J. Pater; 1546782 of Jul. 21, 1925 to L. M. Ford; 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1645534 of Oct. 18, 1927 to O. W. Judkins; 1697464 of Jan. 1, 1929 to W. T. Hofmann; 1754253 of Apr. 15, 1930 to H. C. Avery; 2070571 of Feb. 16, 1937 to M. R. Beasley; **Can. Pats.** 229986 of Apr. 3, 1923 to W. T. Hofmann; 271210 of May 31, 1927 to H. C. Avery; **Brit. Pat.** 237499 of Dec. 31, 1924 to F. H. Clark.

384 [p. 655] **U. S. Pat.** 1952754 of Mar. 27, 1934 to N. P. Harshberger; **Can. Pat.** 359064 of Jul. 14, 1936 to Certain-Teed Products Corp.; 359785 of Aug. 11, 1936 to Building Products, Ltd.

385 [p. 655] **U. S. Pats.** 490668 of Jan. 31, 1893 to G. S. Lee; 1024549 and 1024550 of Apr. 30, 1912 to M. B. Becker; 1113116 of Oct. 6, 1914 to S. H. Goldberg; 1157664 of Oct. 26, 1915 to M. B. Becker; 1214659 of Feb. 6, 1917 to W. P. Dun Lany; 1218217 of Mar. 6, 1917 to E. J. Schroder; 1222594 of Apr. 17, 1917 to M. B. Becker; Design 51438 of Oct. 30, 1917 to A. J. Caron; 1392323 of Oct. 4, 1921 to S. H. Goldberg; 1444550 of Feb. 6, 1923 to R. C. Neptune; 1469606 of Oct. 2, 1923 to C. E. Rahr and Lester Kirschbraun; 1502003 of Jul. 22, 1924 to Herbert Abraham; 1623189 of Apr. 5, 1927 to Lester Kirschbraun; 2036329 of Apr. 7, 1936 to J. D. Giles; **Can. Pat.** 330053 of Feb. 7, 1933 to Patent & Licensing Corp.

386 [p. 655] **U. S. Pats.** 1450712 of Apr. 3, 1923 to W. H. Cady; 1469606 of Oct. 2, 1923 to C. E. Rahr and Lester Kirschbraun; 1623189 of Apr. 5, 1927 to Lester Kirschbraun and W. H. Cady.

387 [p. 657] **U. S. Pats.** 1144313 of Jun. 22, 1915 to S. G. Wright; 1157664 of Oct. 26, 1915 to M. B. Becker; 2013332 of Sep. 3, 1935 to Alfred Anderson; **Can. Pats.** 147910, 147911 and 147912 of May 13, 1913 to M. B. Becker.

388 [p. 657] **U. S. Pats.** 1024550 of Apr. 30, 1912 to M. B. Becker; 1174960 of Mar. 14, 1916 to M. B. Becker; 1181827 of May 2, 1916 to C. S. Bird; 1268105 of Jun. 14, 1918 to S. M. Ford; Design 52538 of Oct. 8, 1918 to T. J. Lords; 1296114 of Mar. 4, 1919 to R. P. Perry; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1472227 of Oct. 30, 1923 to F. C. Overbury; 1540944 of Jun. 9, 1925 to O. D. McFarland.

389 [p. 657] **U. S. Pats.** 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; 1157665 of Oct. 26, 1915 to M. B. Becker; 2056273, 2056274 and 2056275 of Oct. 6, 1936 to R. A. Holdsworth; **Can. Pat.** 330883 of Mar. 14, 1933 to Barrett Co.

390 [p. 657] **U. S. Pats.** 702614 of Jun. 17, 1902 to W. H. Bache; 800320 of Sep. 26, 1905 to T. F. Odell; 1182415 of May 9, 1916 to F. C. Overbury and H. C. Platts; 1203598 of Nov. 7, 1916 to S. M. Ford; 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1449745 of Mar. 27, 1923 to A. L. Clapp; 1491798 of Apr. 29, 1924 to N. P. Harshberger; 1546782 of Jul. 21, 1925 to S. M. Ford; **Can. Pats.** 179755 and 179756 of Oct. 16, 1917 to S. M. Ford; 233543 of Aug. 14, 1923 to A. L. Clapp; **Ger. Pat.** 130634 of Jun. 26, 1901 to H. N. Hansen.

391 [p. 657] **U. S. Pats.** 154843 of Sep. 8, 1874 to Rowell Colby; 449646 of Mar. 31, 1891 to J. A. Smith; 490668 of Jan. 31, 1893 to G. S. Lee; 838232 of Dec. 11, 1906 to J. O. Ballentine; 1126114 of Jan. 26, 1915 to A. S. Spiegel; **Can. Pat.** 105023 of Apr. 30, 1907 to J. O. Ballentine.

392 [p. 657] **U. S. Pat.** 1376215 of Apr. 26, 1921 to C. C. Millard; **Austrian Pat.** 65252 of Dec. 15, 1913 to Heinrich Schwarz.

393 [p. 657] **U. S. Pat.** 1730065 of Oct. 1, 1929 to A. C. Fischer.

394 [p. 658] **U. S. Pats.** 702614 of Jun. 17, 1902 to W. H. Bache; 742614 of Oct. 27, 1903 to J. L. M. Du Four; 875099 of Dec. 31, 1907 to F. C. Overbury; 875595 of Dec. 31, 1907 to F. C. Overbury; 876009 of Jan. 7, 1908 to F. C. Overbury; 881023 of Mar. 3, 1908 to F. C. Overbury; 881024 of Mar. 3, 1908 to F. C. Overbury; 891500 and 891501 of Jun. 23, 1908 to F. C. Overbury; 908125 of Dec. 29, 1908 to F. C. Overbury; 942660 of Dec. 7, 1909 to F. C. Overbury; 966178 of Aug. 2, 1910 to J. L. M. Du Four; 978333 and 978334 of Dec. 13, 1910

to F. C. Overbury; 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1102608 of Jul. 7, 1914 to F. C. Overbury; 1110238 of Sep. 8, 1914 to A. S. Spiegel; 1126932 of Feb. 2, 1915 to Herbert Abraham; 1130368 of Mar. 2, 1915 to R. W. Bird; 1164422 of Dec. 14, 1915 to A. S. Spiegel; 1302414 of Apr. 29, 1919 to C. C. Nerness; 1345099 of Jun. 29, 1920 to F. C. Overbury; 1433983 of Oct. 31, 1922 to Frank Christenson; 1550299 of Aug. 18, 1925 to Arthur Winding; 1583977 of May 11, 1926 to H. G. Kelly; 1596272 of Aug. 17, 1926 to G. M. Jordan; 1597135 of Aug. 24, 1926 to Lester Wittenberg; 1683016 of Sep. 4, 1928 to E. L. Bell and O. M. Beebe; 1722760 of Jul. 30, 1919 to R. C. Neptune; **Can. Pats.** 119710 of Jul. 27, 1909 to Flintkote Mfg. Co.; 217360 of Mar. 28, 1922 to Roofing Patents Co.; **Ger. Pat.** 226401 of Dec. 25, 1907 to Flintkote Mfg. Co.; **Austrian Pat.** 48947 of Jul. 10, 1911 to Heinrich Schwarz.

395 [p. 658] **U. S. Pats.** 1778903 of Oct. 21, 1930 to R. B. Levis; 1834998 of Dec. 8, 1931 to M. R. Becker; 1860047 of May 24, 1932 to A. E. F. Moone; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1923946 of Aug. 22, 1933 to A. E. F. Moone; Reissue 18919 of Aug. 22, 1933 to R. B. Levis; 1928835 of Oct. 3, 1933 to R. B. Levis; 1959986 of May 22, 1934 to A. E. F. Moone; Reissue 19903 of Mar. 24, 1936 to N. P. Harshberger.

396 [p. 658] **U. S. Pats.** 1126932 of Feb. 2, 1915 to Herbert Abraham; 1314476 and 1314477 of Aug. 26, 1919 to F. C. Overbury; 1482090 of Jan. 29, 1924 to F. C. Overbury; 1599512 of Sep. 14, 1926 to W. H. Cady; 1612776 of Dec. 28, 1926 to Lester Kirschbraun; 1751945 of Mar. 25, 1930 to F. C. Overbury; 1752454 of Apr. 1, 1930 to F. C. Overbury; 1755049 of Apr. 15, 1930 to W. H. Cady; 1760873 of Jun. 3, 1930 to Lester Kirschbraun; 1765800 of Jun. 24, 1930 to F. C. Overbury; 1781877 of Nov. 18, 1930 to H. L. Levin; 1782535 and 1782536 of Nov. 25, 1930 to Lester Kirschbraun; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1802032 of Apr. 21, 1931 to F. C. Overbury; 1834954 of Dec. 8, 1931 to Lester Kirschbraun; 1841296 of Jan. 12, 1932 to F. C. Overbury; 1842448 of Jan. 26, 1932 to C. R. Eckert; 1872603 of Aug. 16, 1932 to F. C. Overbury; 1912986 of Jun. 6, 1933 to Lester Kirschbraun; 1927824 of Sep. 26, 1933 to C. R. Eckert; 1950032 of Mar. 6, 1934 to Lester Kirschbraun; 1972133 of Sep. 4, 1934 to M. S. Darrow; 1982215 of Nov. 27, 1934 to Lester Kirschbraun; 2000226 of May 7, 1935 to L. A. Fry; 2011098 of Aug. 13, 1935 to J. L. Wettlaufer; 2048597 of Jul. 21, 1936 to W. H. Cady; 2063268 of Dec. 8, 1936 to J. H. Plunkett; **Can. Pats.** 203981 of Sep. 14, 1920 to Flintkote Co.; 269087 of Mar. 15, 1927 to Flintkote Co.; 292771 of Sep. 3, 1929 to Flintkote Co.; 295373 of Dec. 3, 1929 to Flintkote Co.; 311967 of Jun. 2, 1931 to Patent & Licensing Corp.; 313814 of Jul. 28, 1931 to Patent & Licensing Corp.; 315677 of Sep. 29, 1931 to Flintkote Co.; 316776 of Nov. 3, 1931 to Patent & Licensing Corp.; 330052 of Feb. 7, 1933 to Patent & Licensing Corp.; 354299 of Nov. 19, 1935 to Patent & Licensing Corp.

397 [p. 658] **U. S. Pat.** 1748981 of Mar. 4, 1930 to W. A. Harris.

398 [p. 658] **U. S. Pat.** 1805292 of May 12, 1931 to F. A. Mosher.

399 [p. 658] **U. S. Pats.** 877019 of Jan. 21, 1908 to J. W. Troeger; 1014424 of Jan. 9, 1912 to J. W. Troeger; 1197307 of Sep. 5, 1916 to F. M. Ruschhaupt; 1273388 of Jul. 23, 1918 to J. C. Loyer and O. M. Loyer; 1294785 of Feb. 18, 1919 to S. M. Ford; 1310082 of Jul. 15, 1919 to H. G. Hose; 1352931 of Sep. 14, 1920 to Oscar Altpeter; 1412828 of Apr. 18, 1922 to B. C. Beckman and A. O. Herzog; 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1604941 of Oct. 26, 1926 to W. T. Hofmann; 1662655 of Mar. 13, 1928 to Herbert Abraham; 1716072 of Jun. 4, 1929 to A. A. MacCubbin; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1821552 of Sep. 1, 1931 to Lester Kirschbraun; 1851300 of Mar. 29, 1932 to B. C. Beckman; 1886456 of Nov. 8, 1932 to J. L. Wettlaufer; 1900940 of Mar. 14, 1933 to Lester Kirschbraun; 1984218 of Dec. 11, 1934 to Lester Kirschbraun; 2003699 of Jun. 4, 1935 to Thomas Robinson; **Can. Pats.** 121520 of Oct. 26, 1909 to J. W. Troeger; 213049 of Aug. 23, 1921 to Beckman-Dawson Roofing Co.; 223166 of Aug. 29, 1922 to O. A. Altpeter; 234293 of Sep. 18, 1923 to Steven Troy; 239497 and 239498 of Apr. 22, 1924 to W. T. Hofmann; 270345 of May 3, 1927 to Flintkote Co.; 311314 of May 12, 1931 to Patent & Licensing Corp.; 319983 of Feb. 23, 1932 to Patent & Licensing Corp.; 327488 of Nov. 8, 1932 to Patent & Licensing Corp.; 341331 of May 1, 1934 to Patent & Licensing Corp.

400 [p. 658] **U. S. Pats.** 1256508 of Feb. 19, 1918 to M. B. Becker; 1368947 of Feb. 15, 1921 to W. W. Levis; 1722702 of Jul. 30, 1929 to Lester Kirschbraun and O. A. Heppes;

1756989 of May 6, 1930 to F. C. Overbury; 1760873 of Jun. 3, 1930 to Lester Kirschbraun; 1781877 of Nov. 30, 1930 to H. L. Levin; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1801245 of Apr. 14, 1931 to E. L. Chamberlain; 1807918 of Jun. 2, 1931 to Lester Kirschbraun and O. A. Heppes; 1865579 of Jul. 5, 1932 to C. R. MacDonald; 1873209 of Aug. 23, 1932 to C. R. MacDonald; 1912986 of Jun. 6, 1933 to Lester Kirschbraun; 1913475 of Jun. 13, 1933 to R. W. Conant; 1927824 of Sep. 26, 1933 to C. R. Eckert; 1955131 of Apr. 17, 1934 to Lester Kirschbraun; 1961005 of May 29, 1934 to H. L. Levin; 1970431 of Aug. 14, 1934 to F. C. Overbury; 1989554 of Jan. 29, 1935 to Lester Kirschbraun and O. A. Heppes; 2013351 and 2013352 of Sep. 3, 1935 to H. L. Levin; 2013349 of Sep. 3, 1935 to Lester Kirschbraun; 2015929 of Oct. 1, 1935 to Ernest Goodwin; 2041419 of May 19, 1936 to Lester Kirschbraun; 2043545 of Jun. 9, 1936 to Lester Kirschbraun; 2053723 of Sep. 8, 1936 to Lester Kirschbraun; 2058578 of Oct. 27, 1936 to C. R. Eckert; 2071229 of Feb. 16, 1937 to O. A. Heppes and Lester Kirschbraun; 2074684 of Mar. 23, 1937 to C. R. Eckert; **Can. Pats.** 283901 of Oct. 9, 1928 to Building Products Co., Ltd.; 287870 of Mar. 12, 1929 to Building Products Co., Ltd.; 287889 of Mar. 12, 1929 to Flintkote Co.; 290807 of Jun. 25, 1929 to Flintkote Co.; 295372 of Dec. 3, 1929 to Flintkote Co.; 298268 of Mar. 11, 1930 to Patent & Licensing Corp.; 302605 of Jul. 29, 1930 to Patent & Licensing Corp.; 306999 of Dec. 23, 1930 to Patent & Licensing Corp.; 311967 of Jun. 2, 1931 to Patent & Licensing Corp.; 323863 of Jul. 5, 1932 to Bird & Son, Inc.; 329789 of Jan. 31, 1933 to Barrett Co.; 330052 of Feb. 7, 1933 to Patent & Licensing Corp.; 331511 of Apr. 4, 1933 to Patent & Licensing Corp.; 341267 of May 1, 1934 to Barrett Co.; 342753 of Jul. 3, 1934 to Brantford Roofing Co., Ltd.; 346638 of Dec. 11, 1934 to Barrett Co.; 348281 of Feb. 19, 1935 to Barrett Co.; 353595 of Oct. 15, 1935 to Patent & Licensing Corp.; 359594 of Aug. 4, 1936 to Patent & Licensing Corp.; 360116 and 360117 of Aug. 25, 1936 to Patent & Licensing Corp.

401 [p. 658] **U. S. Pats.** 1850680 of Mar. 22, 1932 to H. L. Levin; 1873886 of Aug. 23, 1932 to G. P. Heppes; 2009617 of Jul. 30, 1935 to N. P. Harshberger; **Can. Pats.** 311312 and 311313 of May 12, 1931 to Patent & Licensing Corp.

402 [p. 658] **U. S. Pats.** 1365800 of Jan. 18, 1921 to E. R. Snyder; Reissue 15352 of May 9, 1922 to E. R. Snyder; 1516243 of Nov. 18, 1924 to R. P. Perry; 1612776 of Dec. 28, 1926 to Lester Kirschbraun; 1742724 of Jan. 7, 1930 to R. P. Perry; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1802032 of Apr. 21, 1931 to F. C. Overbury; 1864806 of Jun. 28, 1932 to D. A. Cumfer; 1870426 of Aug. 9, 1932 to G. J. Snyder; 1873212 of Aug. 23, 1932 to F. C. Overbury; 1890017 of Dec. 6, 1932 to C. R. MacDonald; 1890018 of Dec. 6, 1932 to F. C. Overbury; 1961166 of Jun. 5, 1934 to C. E. Rahr; 1967856 of Jul. 24, 1934 to B. C. Beckman; **Can. Pats.** 274752 of Oct. 18, 1927 to Flintkote Co.; 287509 and 287510 of Feb. 26, 1929 to Flintkote Co.; 288077 of Mar. 19, 1929 to Flintkote Co.; 304824 of Oct. 14, 1930 to Flintkote Co.; 313814 of Jul. 28, 1931 to Patent & Licensing Corp.; 316776 of Nov. 3, 1931 to Patent & Licensing Corp.; 331511 of Apr. 4, 1933 to Patent & Licensing Corp.

403 [p. 658] **U. S. Pat.** 1673991 of Jun. 19, 1928 to F. C. Overbury; **Can. Pat.** 260003 of Apr. 20, 1926 to F. C. Overbury.

404 [p. 658] **U. S. Pats.** 320979 of Jun. 30, 1885 to L. B. Snow; 394033 of Dec. 4, 1888 to S. E. Trott; 877019 of Jan. 21, 1908 to J. W. Troeger; 886912 of May 5, 1908 to C. W. Young and J. G. Burruss; 933221 of Sep. 7, 1909 to C. W. Young; 1048517 of Dec. 31, 1912 to J. G. Fox and H. W. Simms; 1053792 of Feb. 18, 1913 to J. F. Dietz; 1191932 of Jul. 18, 1916 to J. C. Loyer and O. M. Loyer; Reissue 14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1763601 of Jun. 10, 1930 to Lester Kirschbraun.

405 [p. 658] **U. S. Pats.** 1104998 of Jul. 28, 1914 to F. C. Overbury; 1108884 of Sep 1, 1914 to C. S. Bird; 1153418 of Sep. 14, 1915 to C. S. Bird; 1184509 of May 23, 1916 to C. S Bird; 1897139 of Feb. 14, 1933 to F. C. Overbury.

406 [p. 658] **U. S. Pats.** 1585692 and 1585693 of May 25, 1926 to Thomas Robinson; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1676351 of Jul. 10, 1928 to Thomas Robinson; 1698242 of Jan. 8, 1929 to Thomas Robinson; 1716505 and 1716056 of Jun. 11, 1929 to Thomas Robinson; 1765796 of Jun. 24, 1930 to Lester Kirschbraun; 1807435 of May 26, 1931 to Thomas Robinson; 1818009 of Aug. 11, 1931 to Thomas Robinson; 1819199 and

1819200 of Aug. 18, 1931 to Thomas Robinson; 1820834 of Aug. 25, 1931 to Thomas Robinson; 1863880 of Jun. 21, 1932 to Thomas Robinson; 1868139 of Jul. 19, 1932 to D. D. Forbes; 1873040 of Aug. 23, 1932 to Thomas Robinson; 1909318 of May 16, 1933 to Thomas Robinson; 1922501 of Aug. 15, 1933 to Thomas Robinson; 1941491 of Jan. 2, 1934 to Thomas Robinson; 1952828 of Mar. 27, 1934 to C. E. Volkhardt; 1973931 of Sep. 18, 1934 to Thomas Robinson; 2035921 of Mar. 31, 1946 to R. G. Quinn; 2057167 of Oct. 13, 1936 to J. C. Sherman; **Can. Pats.** 158954 of Nov. 17, 1914 to Herbert Abraham; 175623 of Mar. 13, 1917 to J. C. Loyer and O. M. Loyer; 286503 of Jan. 15, 1929 to Flintkote Co.; 301468 of Jun. 24, 1930 to Lancaster Asphalt, Inc.; 315944 of Oct. 6, 1931 to Lancaster Asphalt, Inc.; 316128 of Oct. 13, 1931 to Lancaster Asphalt, Inc.; 318879 of Jan. 12, 1932 to Lancaster Asphalt, Inc.; 322097 of May 3, 1932 to Lancaster Asphalt, Inc.; 323293 and 323294 of Jun. 14, 1932 to Lancaster Asphalt, Inc.

407 [p. 658] **U. S. Pat.** 1638746 of Aug. 9, 1927 to Thomas Robinson.

408 [p. 658] **U. S. Pats.** 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; 1368947 of Feb. 15, 1921 to W. W. Levis.

409 [p. 659] **U. S. Pat.** 1758059 of May 13, 1930 to C. E. Rahr.

410 [p. 659] **U. S. Pat.** 1340347 of May 18, 1920 to C. E. Rahr.

411 [p. 659] **U. S. Pats.** 1365800 of Jan. 18, 1921 to E. R. Snyder; Reissue 15352 of May 9, 1922 to E. R. Snyder; 1516243 of Nov. 18, 1924 to R. P. Perry; 1758059 of May 13, 1930 to C. E. Rahr; 1802032 of Apr. 21, 1931 to F. C. Overbury; **Can. Pats.** 225117 of Oct. 24, 1922 to Barrett Co.; 279136 of Apr. 3, 1928 to Flintkote Co.

412 [p. 659] **U. S. Pats.** 1270654 of Jan. 25, 1918 to F. C. Overbury; 1452978 of Apr. 24, 1923 to J. S. Miller, Jr.; **Can. Pat.** 185435 of Jul. 9, 1918 to F. C. Overbury; **Brit. Pat.** 138879 of Sep. 3, 1914 to Flintkote Co.

413 [p. 659] See Reference 380.

414 [p. 659] **U. S. Pat.** 1583563 of May 4, 1926 to Herbert Abraham; **Can. Pat.** 291769 of Jul. 30, 1929 to Duracolor Co.

415 [p. 659] **U. S. Pats.** 1445777 of Feb. 20, 1923 to T. B. Lehon; 1518988 of Dec. 9, 1924 to T. B. Lehon; 1759901 of May 27, 1930 to N. P. Harshberger; **Can. Pats.** 126321 of Jun. 14, 1910 to G. H. Pedlar; 238327 and 238328 of Jan. 23, 1923 to Lehon Co.

416 [p. 659] **U. S. Pats.** 1361517 of Dec. 7, 1920 to A. T. Cavey; 1429728 of Sep. 19, 1922 to C. N. Forrest; 1447290 of Mar. 6, 1923 to A. C. Fischer; 1448155 of Mar. 13, 1923 to Karl Schutte; 1494380 of May 20, 1924 to Karl Schutte; 1550310 of Aug. 18, 1925 to A. C. Fischer; 1743764 of Jan. 14, 1930 to A. C. Fischer; 1769632, 1769633 and 1769634 of Jul. 1, 1930 to A. C. Fischer; 1789284, 1789285, 1789286 and 1789287 of Jan. 13, 1931 to A. C. Fischer; 1861408 of May 31, 1932 to A. C. Fischer; 1881435 of Oct. 11, 1932 to A. C. Fischer; 1924047 of Aug. 22, 1933 to A. C. Fischer; 1939004 of Dec. 12, 1933 to A. C. Fischer; 2065439 of Dec. 22, 1936 to A. C. Fischer; **Can. Pats.** 214157 of Nov. 8, 1921 to A. T. Cavey; 233126 of Jul. 31, 1923 to Barber Asphalt Co.; **Brit. Pats.** 156804 of Nov. 17, 1921 to A. T. Cavey; 353910 of Apr. 29, 1930 to Durastic Bituminous Products, Ltd.

417 [p. 659] **U. S. Pat.** 1447750 of Mar. 6, 1923 to C. S. Bird.

418 [p. 659] **U. S. Pats.** 1448155 of Mar. 13, 1923 to Karl Schutté; 1494380 of May 20, 1924 to Karl Schutté; **Can. Pat.** 249586 of May 12, 1925 to Karl Schutté.

419 [p. 659] **U. S. Pat.** 1810880 of Jun. 16, 1931 to A. C. Fischer.

420 [p. 659] "Neuere Dach-, Fuszboden- und Wandbeläge mit Bitumenverwendung," by Walter Obst, *Teer u. Bitumen*, 33, 27 (1935); **U. S. Pats.** 19627 of Mar. 16, 1858 to W. T. de Golyer; 26868 of Jan. 17, 1860 to Hiram Tucker; 136722 of Mar. 11, 1875 to S. C. Hogue; 137566 of Apr. 8, 1873 to E. R. Percy; 140945 of Jul. 15, 1873 to Charles Mueller; 191208 of May 22, 1877 to C. M. Warren; 252524 of Jan. 17, 1882 to L. L. Sagendorph; 255088 of Mar. 14, 1882 to L. L. Sagendorph; 825870 of Jul. 10, 1906 to Julius Schirra; 1138069 of May 4, 1915 to Addison Applegate; 1518337 of Dec. 9, 1924 to J. F. Makowski; 1592807 of Jul. 13, 1926 to C. N. Forrest; 1627531 of May 3, 1927 to F. A. Browne; 1700930 of Feb. 5, 1929 to C. N. Forrest; 1707255 of Apr. 2, 1929 to M. S. Darrow; 1913666 and 1913667 of Jun. 13, 1933 to N. P. Harshberger; 2044781, 2044782, 2044783, 2044784, 2044785 and 2044786 of

Jun. 23, 1936 to N. P. Harshberger; 2044787 and 2044788 of Jun. 23, 1936 to N. P. Harshberger and S. A. Ochs; 2068396 of Jan. 19, 1937 to S. W. Chaffee; **Can. Pats.** 339345 and 339346 of Feb. 13, 1934 to Bakelite Building Products Co., Inc; 363123, 363124, 363125 and 363126 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.; **Ger. Pat.** 212527 of Oct. 30, 1926 to Ludwig Esselborn.

421 [p. 659] **Can. Pat.** 349310 of Apr. 2, 1935 to H. H. Robertson Co.

422 [p. 659] **U. S. Pats.** 404542 of Nov. 3, 1863 to L. S. Mills and C. H. Smith; 44821 of Oct. 25, 1864 to Joseph Rodefer; 145705 of Dec. 16, 1873 to Horace Wheeler; 175533 of Mar. 28, 1876 to C. M. Warren; 357503 of Feb. 8, 1887 to William Redett; 396292 of Jan. 15, 1899 to E. C. Richmond; 756180 of Mar. 29, 1904 to J. H. Munro; 769624 of Sep. 6, 1904 to J. H. Munro; 769663 and 769664 of Sep. 6, 1904 to J. H. Munro; 802771 of Oct. 24, 1905 to J. H. Munro; 967542 of Aug. 16, 1910 to J. H. Munro; 986140 of Feb. 28, 1911 to Hedley Britton; 1134217 of Apr. 6, 1915 to J. H. Munro; 1173678 of Feb. 29, 1916 to J. H. Munro; 1217152 of Feb. 27, 1917 to Albert Chauvin; 1229622 of Jun. 12, 1917 to V. F. Lake; 1250622 and 1250623 of Dec. 18, 1917 to J. H. Munro; 1439072 of Dec. 19, 1922 to G. R. Dean; 1439434 and 1439435 of Dec. 19, 1922 to J. H. Munro; Reissue 15820 of Apr. 15, 1924 to J. H. Munro; 1549263 of Aug. 11, 1925 to R. T. Johnston; 1558186 of Oct. 20, 1925 to V. F. Lake; 1561581 of Nov. 17, 1925 to Herbert Abraham; 1583163 of May 4, 1926 to J. H. Munro; 1588540 of Jun. 15, 1926 to A. B. Fosseen; 1590385 of Jun. 29, 1926 to V. F. Lake; 1593094, 1593095 and 1593096 of Jul. 20, 1926 to J. H. Munro; 1728795 of Sep. 17, 1929 to J. H. Griffin; 1852696 of Apr. 5, 1932 to S. W. Chaffee; 1854512 of Apr. 19, 1932 to O. A. Heppes; 1936055 of Nov. 21, 1933 to Donald Heaney; 1937255 of Nov. 28, 1933 to H. P. Taylor; Reissue 19518 of Apr. 9, 1935 to S. W. Chaffee; **Can. Pats.** 95338 of Oct. 3, 1905 to J. H. Munro; 141514 of Jul. 2, 1912 to J. H. Munro; 244144 and 244145 of Nov. 4, 1924 to J. H. Munro; 246520 of Feb. 3, 1925 to J. H. Munro; 247684 of Mar. 17, 1925 to J. H. Munro; 248634 of Apr. 14, 1925 to J. H. Munro; 248716 of Apr. 14, 1925 to Ruberoid Co., Ltd.; 248719 and 248720 of Apr. 14, 1925 to Slate Veneer Co.; 253852 of Sep. 22, 1925 to J. H. Munro; 328693 of Dec. 20, 1932 to Patent & Licensing Corp.; **Brit. Pat.** 241732 of Nov. 17, 1924 to Herbert Abraham; **Ger. Pat.** 129850 of Jun. 12, 1901 to M. F. Siegs; **Danish Pat.** 19843 of Feb. 5, 1915 to A. Nielson.

423 [p. 659] **U. S. Pats.** 113882 of Apr. 18, 1871 to T. N. Hickcox; 153749 of Aug. 4, 1874 to Rowell Colby; 164749 of Jun. 22, 1875 to Delaplaine McDaniel; 632691 of Sep. 12, 1899 to W. C. Bates; 836157 of Nov. 20, 1906 to P. W. Turner; 845290 of Feb. 26, 1907 to E. H. Binns; 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1076111 of Oct. 21, 1913 to F. H. Grant; 1094893 of Apr. 28, 1914 to F. H. Grant; 1354050 of Sep. 28, 1920 to Arthur Lovell; 1512248 of Oct. 21, 1924 to James Smith; 1513940 of Nov. 4, 1924 to James Smith; 1536550 of May 5, 1925 to J. H. Young; 1551317 and 1551318 of Aug. 25, 1925 to John Logan; 1589841 of Jun. 22, 1926 to J. A. Daly; 1593205 of Jul. 20, 1926 to J. H. Young; 1636388 of Jul. 19. 1927 to Thomas Robinson; 1638746 of Aug. 9, 1927 to Thomas Robinson; 1663565 of Mar, 27, 1928 to Thomas Robinson; 1665222 of Apr. 10, 1928 to Thomas Robinson; 1666755 of Apr. 17, 1928 to C. E. Rahr and F. C. Overbury; 1673585 of Jun. 12, 1928 to F. C. Overbury; 1698267 of Jan. 8, 1929 to Lester Kirschbraun; 1701918 of Feb. 12, 1929 to J. H. Gillis; 1701926 of Feb. 12, 1929 to Lester Kirschbraun; 1723076 of Aug. 6, 1929 to R. S. Reynolds; 1727132 of Sep. 3, 1929 to R. S. Reynolds; 1753303 of Apr. 8, 1930 to R. S. Reynolds; 1794674 of Mar. 3, 1931 to D. A. Cumfer; 1813084 of Jul. 7, 1931 to Thomas Robinson; 1813089 of Jul. 7, 1931 to W. M. Shakespeare; 1819270 of Aug. 8, 1931 to W. M. Shakespeare; 1822410 of Sep. 8, 1931 to H. C. Macan; 1826761 of Oct. 13, 1931 to D. F. Fradette; 1829886 of Nov. 3, 1931 to C. E. Yates and H. C. Macan; 1864055 of Jun. 21, 1932 to W. H. Finkeldey; 1865959 of Jul. 5, 1932 to T. C. Prouty; 1871067 and 1871068 of Aug. 9, 1932 to H. C. Macan; 1908127 of May 9, 1933 to W. T. Deacon; 1909862 of May 16, 1933 to I. C. Honegger; 1927586 of Sep. 19, 1933 to D. F. Fradette; 2016429 of Oct. 8, 1935 to H. P. Hayden; 2032275 of Feb. 25, 1936 to D. F. Fradette; 2043271 of Jun. 9, 1936 to J. H. Wahl and P. H. Doe; 2050248 of Aug. 11, 1936 to J. B. Eisen; **Can. Pats.** 152970 of Jan. 6, 1914 to F. H. Grant; 227922 of Jan. 16, 1923 to E. A. Bradshaw; 236658 of Dec. 25, 1923 to H. H. Robertson Co.; 249326 of May 5, 1925 to Anaconda Sales Co.; 249558 of May 12, 1925 to Anaconda Sales

Co.; 253276 of Sep. 1, 1925 to J. H. Gillis; 255122 of Nov. 3, 1925 to Anaconda Sales Co.; 255725 and 255760 of Nov. 24, 1925 to Anaconda Sales Co.; 261530 of Jun. 8, 1926 to Anaconda Sales Co.; 285451 of Dec. 11, 1928 to I. N. Lewis; 309214 of Mar. 10, 1931 to Anaconda Sales Co.; 311037 and 311038 of May 5, 1931 to Anaconda Sales Co.; 324387 of Jul. 19, 1932 to Singmaster & Breyer, Inc.; 327879 of Nov. 22, 1932 to H. H. Robertson Co.; 364986 of Mar. 23, 1937 to D. F. Fradette; **Brit. Pats.** 215953 of May 8, 1923 to H. H. Robertson Co.; 220644 of Aug. 15, 1924 to International Copperclad Co.; 220646 of Aug. 15, 1924 to J. H. Gillis; 220647 of Aug. 15, 1924 to International Copperclad Co.; 307144 of Dec. 24, 1927 to J. A. Montgomerie; 340168 of Feb. 17, 1930 to International Copperclad Co.; 415416 of Feb. 27, 1933 to Hermann von Forster; 415739 of Feb. 27, 1933 to Hermann von Forster; 446943 of May 8, 1936 to N. V. Internationale Alfol Maatschappij; **Ger. Pats.** 249986 of Aug. 30, 1910 to Naumann Schefftel; 566110 of Feb. 26, 1930 to International Copperclad Co.; Appl. M-110358 of May 25, 1929 to Vereinigte Deutsche Metallwerke, A.-G.; Appl. M-112940 of Nov. 30, 1929 to Vereinigte Deutsche Metallwerke, A.-G.; Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.; Design 1213311 of Mar. 2, 1932 to Carl Rahtkens.

424 [p. 659] **U. S. Pats.** 409096 of Aug. 13, 1889 to Alois Blank; Reissue 11106 of Aug. 26, 1890 to Alois Blank; 1449058 of Mar. 20, 1923 to Thomas Robinson; 1559040 and 1559041 of Oct. 27, 1925 to J. H. Gillis; 1567048 of Dec. 29, 1925 to J. H. Gillis; 1574385 of Feb. 23, 1926 to J. H. Gillis; 1574446 of Feb. 23, 1926 to Thomas Robinson; 1589636 and 1589637 of Jun. 22, 1926 to J. H. Gillis; 1589841 of Jun. 22, 1926 to J. A. Daly; 1612885 of Jan. 4, 1927 to Thomas Robinson; 1625888 of Apr. 26, 1927 to J. H. Gillis; 1680096 of Aug. 7, 1927 to R. E. Drake; 1700774 of Feb. 5, 1929 to C. E. Rahr and R. E. Drake; 1701918 of Feb. 12, 1929 to J. H. Gillis; 1720708 of Jul. 16, 1929 to J. H. Young; 1753721 of Apr. 8, 1930 to Thomas Robinson; 1794449 of Mar. 3, 1931 to R. E. Drake; 1794748 of Mar. 3, 1931 to C. E. Yates; 1805920 of May 19, 1931 to Fred Muschler; 1852169 of Apr. 5, 1932 to Solomon Levy; 1871105 of Aug. 9, 1932 to C. E. Yates and H. C. Macan; 2042030 of May 26, 1936 to U. C. Tainton; **Can. Pats.** 39099 of Jun. 10, 1892 to R. G. Westphalen; 247312 of Mar. 3, 1925 to Anaconda Sales Co.; 253276 of Sep. 1, 1925 to J. H. Gillis; 267672 of Jan. 18, 1927 to Anaconda Sales Co.; 269401 of Mar. 29, 1927 to Anaconda Sales Co.; 278534 of Mar. 13, 1928 to Anaconda Sales Co.; 280921 of Jun. 12, 1928 to Anaconda Sales Co.; 312117 of Jun. 9, 1931 to Anaconda Sales Co.; 323649 of Jun. 28, 1932 to Anaconda Sales Co.; **Brit. Pats.** of 1889 (Mar. 27), 13329 to Alois Blank; 186363 of Mar. 29, 1921 to M. Höchstädter; 196063 of Jan. 12, 1922 to A. I. G. Warren; 199009 of May 15, 1923 to Roofing Research & Engineering Corp.; 211478 of Feb. 13, 1924 to Anaconda Sales Co.; 220647 of Aug. 15, 1924 to International Copperclad Co.; 278437 of Jul. 7, 1926 to A. I. G. Warren; 324487 of Jan. 15, 1929 to W. M. Shakespeare; 349690 of Mar. 22, 1930 to International Copperclad Co.; 368463 of Jan. 17, 1931 to International Copperclad Co.; **Ger. Pats.** 43349 of May 25, 1888 to Arthur Siebel; 430641 of Feb. 10, 1924 to International Copperclad Co.; 489103 of Jul. 10, 1926 to Flintkote Co.; Appl. J-23.30 of Mar. 21, 1930 to International Copperclad Co.; Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.

425 [p. 659] **U. S. Pats.** 1488186 of Mar. 25, 1924 to J. H. Young; 1589841 of Jun. 22, 1926 to J. A. Daly; 1593205 of Jul. 20, 1926 to J. H. Young; 1720708 of Jul. 16, 1929 to J. H. Young; 1955572 of Apr. 17, 1934 to Jacob Adler and Paul Doerseln; **Can. Pat.** 349202 of Apr. 2, 1935 to Jacob Adler and Paul Doerseln; **Brit. Pats.** 190470 of Dec. 4, 1922 to H. H. Robertson Co.; 192377 and 192378 of Jan. 15, 1923 to H. H. Robertson Co.; **Ger. Pat.** 411273 of Jan. 18, 1923 to H. H. Robertson Co.

426 [p. 659] **U. S. Pat.** 1521128 of Dec. 30, 1924 to Thomas Robinson.

427 [p. 659] **U. S. Pat.** 2060083 of Nov. 10, 1936 to R. T. Johnston; **Can. Pats.** 362806 and 362807 of Dec. 22, 1936 to Bakelite Corp.

428 [p. 659] **Can. Pat.** 327877 of Nov. 22, 1932 to H. H. Robertson Co.

429 [p. 659] **U. S. Pat.** 845290 of Feb. 26, 1907 to E. H. Binns.

430 [p. 660] **U. S. Pats.** 788358 of Apr. 25, 1905 to F. D. Jacobs; 816661 of Apr. 3, 1906 to F. D. Jacobs; Reissue 12475 of Apr. 24, 1906 to F. D. Jacobs; 845290 of Feb. 26, 1907 to E. H. Binns; 1002301 of Sep. 5, 1911 to E. T. Newsome; 1053792 of Feb. 18, 1913 to J. F. Dietz; 1059682 of Apr. 22, 1913 to T. D. Miller; 1167949 of Jan. 11, 1916 to P. M. Stewart;

Design 49171 of Jun. 13, 1916 to B. S. Annis; 1191932 of Jul. 18, 1916 to J. C. Loyer and O. M. Loyer; 1195090 of Aug. 15, 1916 to H. H. Robertson; 1226564 of May 15, 1917 to T. D. Miller; Reissue 14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1277755 of Sep. 3, 1918 to H. H. Robertson; 1277758 of Sep. 3, 1918 to W. W. Roney; 1358113 of Nov. 9, 1920 to H. H. Robertson; 1471396 of Oct. 23, 1923 to A. A. Griswold; 1481559 of Mar. 11, 1924 to Frank Basel; 1492609 of May 6, 1924 to J. T. Simpson; 1512248 of Oct. 21, 1924 to James Smith and M. E. Constable; 1513940 of Nov. 4, 1924 to James Smith and M. E. Constable; 1516045 of Nov. 18, 1924 to Heinrich Kollbrunner; 1657979 of Jan. 31, 1928 to F. W. Thomas; 1676351 of Jul. 10, 1928 to Thomas Robinson; 1700561 of Jan. 29, 1929 to F. J. Commin; 1814532 of Jul. 14, 1931 to W. A. Sutherland; 1868803 of Jul. 26, 1932 to F. C. Overbury; 1910312 of May 23, 1903 to Frank Young; **Can. Pats.** 185257 of Jul. 2, 1918 to T. D. Miller; 230268 of Apr. 10, 1923 to H. H. Robertson Co.; 233564 of Aug. 14, 1923 to H. H. Robertson Co.; **Brit. Pats.** of 1912 (Jan. 17), 1360 to H. R. Wardell; 106997 and 106998 of Jun. 3, 1916 to H. H. Robertson Co.; 136984 of Mar. 24, 1919 to T. D. Miller; 159864 of Mar. 9, 1922 to H. H. Robertson Co.; 347105 of Jan. 10, 1920 to F. J. Commin and A. H. J. Wright; **Ger. Pat.** Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.

431 [p. 660] **U. S. Pat.** 1826706 of Oct. 13, 1931 to Isadore Davis.

432 [p. 660] **U. S. Pats.** 1358113 of Nov. 9, 1920 to H. H. Robertson; 1716706 of Jun. 11, 1929 to Ami Rey; 1740883 of Dec. 24, 1929 to H. A. Teeple; **Can. Pat.** 163058 of Jun. 8, 1915 to F. H. Grant.

433 [p. 660] **U. S. Pat.** 1765796 of Jun. 24, 1930 to Lester Kirschbraun; **Brit. Pat.** 353910 of Apr. 29, 1930 to Durastic Bituminous Products, Ltd.

434 [p. 660] **U. S. Pats.** 1730065 of Oct. 1, 1929 to A. C. Fischer; 1795913 of Mar. 10, 1931 to W. W. Weaver.

435 [p. 660] **U. S. Pats.** 320979 of Jun. 30, 1885 to L. B. Snow; 394033 of Dec. 4, 1888 to S. E. Trott; 877019 of Jan. 21, 1908 to J. W. Troeger; 886912 of May 5, 1908 to C. W. Young and J. G. Burruss; 889235 of Jun. 2, 1908 to C. B. Jameson; 933221 of Sep. 7, 1909 to C. W. Young; 1048517 of Dec. 31, 1912 to J. G. Fox and H. W. Simms; 1104998 of Jul. 28, 1914 to F. C. Overbury; 1108884 of Sep. 1, 1914 to C. S. Bird; 1153418 of Sep. 14, 1915 to C. S. Bird; 1184509 of May 23, 1916 to C. S. Bird; 1191932 of Jul. 18, 1916 to J. C. Loyer and O. M. Loyer; 1325546 of Dec. 23, 1919 to H. R. Wardell; 1447290 of Mar. 6, 1923 to A. C. Fischer; 1531151 of Mar. 24, 1925 to H. H. Steele; 1585692 and 1585693 of May 25, 1926 to Thomas Robinson; 1629287 of May 17, 1927 to S. K. Milligan; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1644652 of Oct. 4, 1927 to Lester Kirschbraun; 1665450 of Apr. 10, 1928 to H. A. Garber; 1676351 of Jul. 10, 1928 to Thomas Robinson; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; Reissue 17143 of Nov. 20, 1928 to A. C. Fischer; 1698242 of Jan. 8, 1929 to Thomas Robinson; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1716505 and 1716506 of Jun. 11, 1929 to Thomas Robinson; 1742724 of Jan. 7, 1930 to R. P. Perry; 1763601 of Jun. 10, 1930 to Lester Kirschbraun; 1776949 of Sep. 30, 1930 to P. E. Lumbard.

436 [p. 660] **U. S. Pats.** 1261280 of Apr. 2, 1918 to F. C. Overbury; 1296324 of Mar. 4 1919 to J. A. Scharwath; 1325546 of Dec. 23, 1919 to H. R. Wardell; 1689985 of Oct. 30, 1928 to H. R. Wardell; **Can. Pats.** 96636 of Dec. 19, 1905 to W. H. Wooswick; 190354 of May 13, 1919 to H. R. Wardell; 234293 of Sep. 18, 1923 to Steven Troy.

437 [p. 660] **U. S. Pats.** 322990 of Jul. 28, 1885 to Josef Sporny and Ian Zarski; 396292 of Jan. 15, 1889 to E. C. Richmond; 398332 of Feb. 19, 1889 to Julius Jaquet; 985140 of Feb. 28, 1911 to Hedley Button; 1208972 of Dec. 19, 1916 to Richard Jelier; 1403392 of Jan. 10, 1922 to Cecil Davis; 1438302 of Dec. 12, 1922 to Fred Frederiksen; 1480704 of Jan. 15, 1924 to H. A. Wheeler; 1536519 of May 5, 1925 to E. F. Parker; 1558005 of Oct. 20, 1925 to Dozier Finley; 1661562 of Mar. 6, 1928 to G. E. Conway; 2038192 of Apr. 21, 1936 to F. C. Overbury; 2062149 of Nov. 24, 1936 to G. R. Stark and J. A. Main; **Ger. Pat.** Design 1260164 of Mar. 3, 1933 to Robert Beckmann.

438 [p. 660] **U. S. Pats.** 1448614 of Mar. 13, 1923 to A. H. Carrier; 1480704 of Jan. 15, 1924 to H. A. Wheeler; 1953288 of Apr. 3, 1934 to M. L. Caton; 2039776 of May 5, 1936 to M. L. Caton; 2060084 of Nov. 10, 1936 to R. T. Johnston; **Can. Pat.** 312298 of Jun. 16, 1931 to J. A. McKercher; 355646 of Jan. 28, 1936 to International Paper Co.

439 [p. 660] **U. S. Pat.** 1814291 of Jul. 14, 1931 to M. L. Caton; **Can. Pat.** 298017 of Mar. 4, 1930 to Flintkote Co.

440 [p. 660] **U. S. Pat.** 1088803 of Mar. 3, 1914 to G. F. Bishopric; **Can. Pat.** 363039 of Dec. 29, 1936 to Patent & Licensing Corp.

441 [p. 660] **U. S. Pat.** 1871090 of Aug. 9, 1932 to W. M. Shakespeare; 2026608 of Jan. 7, 1936 to S. S. Calafati.

442 [p. 660] **U. S. Pats.** 1887 of Dec. 10, 1840 to William Docker (slate); 55087 of May 29, 1866 to William Green; **Ger. Pats.** 707 of Jul. 4, 1877 to Ludwig Nagel; 100572 of Feb. 27, 1898 to Christian Fleisch; Design 1242227 of Apr. 4, 1932 to G. A. Türke.

443 [p. 661] **Can. Pats.** 297472 and 297473 of Feb. 11, 1930 to Guyton & Cumfer Mfg. Co.

444 [p. 662] **U. S. Pats.** 1497116 of Jun. 10, 1924 to C. M. Olson; 1783023 of Nov. 25, 1930 to S. G. Martin.

445 [p. 662] **U. S. Pats.** 292585 of Jan. 29, 1884 to E. B. Repp (metal); 1077095 of Oct. 28, 1913 to W. S. Orth; 1115866 of Nov. 3, 1914 to H. M. Reynolds; 1159766 of Nov. 9, 1915 to G. P. Heppes; 1172067 and 1172068 of Feb. 15, 1916 to A. S. Spiegel; 1192132 of Jul. 25, 1916 to A. S. Spiegel; 1227776 of May 29, 1917 to W. H. Garges; 1244654 of Oct. 30, 1917 to A. S. Spiegel; 1251704 of Jan. 1, 1918 to A. S. Spiegel; 1285147 of Nov. 19, 1918 to W. A. Harris; 1389979 of Sep. 6, 1921 to C. E. Rahr and Calvin Russell; 1417641 of May 30, 1922 to L. M. Stuffings; 1425991 and 1425992 of Aug. 15, 1922 to E. W. Lesher; 1442379 of Jan. 16, 1923 to Herbert Abraham; 1460827 of Jul. 3, 1923 to A. R. Purdy; 1474380 of Nov. 20, 1923 to Thomas Robinson; 1481193 of Jan. 15, 1924 to A. C. Fischer; 1528471 of Mar. 3, 1925 to C. H. Drumm; 1580755 and 1580756 of Apr. 13, 1926 to J. A. Murray; 1592014 and 1592015 of Jul. 13, 1926 to J. A. Topping; 1601127 of Sep. 28, 1926 to O. D. McFarland; 1602006 of Oct. 5, 1926 to A. C. Fischer; 1631936 of Jun. 7, 1927 to George Ritter; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; 1690396 of Nov. 6, 1928 to L. W. Block; 1719111 of Jul. 2, 1929 to R. A. Holdsworth; 1724269 of Aug. 13, 1929 to Dozier Finley; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; 1972028 of Aug. 28, 1934 to H. T. Nichols; 2064263 of Dec. 15, 1936 to S. A. Kiellar; **Can. Pats.** 152981 of Jan. 6, 1914 to W. S. Orth; 201056 of Jun. 15, 1920 to Flintkote Co.; 210450 of Apr. 12, 1921 to Roofing Patents Co.; 211667 of May 17, 1921 to Roofing Patents Co.; 216870 of Mar. 14, 1922 to Roofing Patents Co.; 220780 of Jul. 11, 1922 to Roofing Patents Co.; 220877 of Jul. 18, 1922 to C. E. Rahr; 223888 of Sep. 19, 1922 to Roofing Patents Co.; 224656 of Oct. 10, 1922 to Guyton & Cumfer Mfg. Co.; 244019 of Oct. 28, 1924 to Guyton & Cumfer Mfg. Co.; 274716 of Oct. 18, 1927 to P. E. Sinnett; 341267 of May 1, 1934 to Barrett Co.

446 [p. 662] **U. S. Pats.** 1295361 of Feb. 25, 1919 to F. C. Overbury; 1345099 of Jun. 29, 1920 to F. C. Overbury; 1366590 of Jan. 25, 1921 to F. C. Overbury; 1560301 of Nov. 3, 1925 to F. C. Overbury; **Can. Pat.** 248538 of Apr. 7, 1925 to Flintkote Co.

447 [p. 662] **U. S. Pats.** 1295361 of Feb. 25, 1919 to F. C. Overbury; 1365947 of Jan. 18, 1921 to F. C. Overbury; 1410903 of Mar. 28, 1922 to G. B. Ferguson; Reissue 15439 of Aug. 29, 1922 to G. B. Ferguson; 1467510 of Sep. 11, 1923 to G. T. Smith, Jr.; 1474380 of Nov. 20, 1923 to Thomas Robinson; 1476745 and 1476746 of Dec. 11, 1923 to E. J. Williams; 1542475 of Jun. 16, 1925 to R. A. Ruess; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; **Can. Pat.** 321137 of Apr. 5, 1932 to C. S. Crary.

448 [p. 663] **U. S. Pats.** 1348498 of Aug. 3, 1920 to C. L. Keller; 1467779 of Sep. 11, 1923 to W. J. Dremann; 1516238 of Nov. 18, 1924 to C. W. Mortimer; 1536027 of Apr. 28, 1925 to C. W. Mortimer; 1591042 of Jul. 6, 1926 to J. W. Ingels; 1614446 of Jan. 11, 1927 to A. R. Lukens; 1631936 of Jun. 7, 1927 to George Ritter; 1683285 of Sep. 4, 1928 to N. Z. Butterick; 1703170 of Feb. 26, 1929 to F. W. Preston; 1888055 of Nov. 15, 1932 to R. J. Tobin and G. A. Tobin; 1911141 of May 23, 1933 to A. C. Fischer; 1924650 of Aug. 29, 1933 to G. B. Payne; 1929165 of Oct. 3, 1933 to William Freegard; 2034602 of Mar. 17, 1936 to A. J. Anderson; 2071430 of Feb. 23, 1937 to F. P. Reynolds; 2072289 of Mar. 2, 1937 to C. A. Barth; **Can. Pats.** 322046 of May 3, 1932 to Bird & Son, Inc.; 356255 of Mar. 3, 1936 to Bakelite Building Products Co., Inc.; 364281 of Feb. 23, 1937 to Building Products, Ltd.

449 [p. 663] **U. S. Pats.** 117601 of Aug. 1, 1871 to C. W. Chaffee; 310192 of Jan. 6, 1885

to J. T. Edson; 430362 of Jun. 17, 1890 to G. H. Babcock; 912057 of Feb. 9, 1909 to E. P. Auger (tile); 1070738 of Aug. 19, 1913 to Calvin Russell; 1115741 of Nov. 3, 1914 to Calvin Russell; 1116149 of Nov. 3, 1914 to A. S. Spiegel; Reissue 14058 of Feb. 8, 1916 to Calvin Russell; 1274605 of Aug. 6, 1918 to Calvin Russell; 1645767 of Oct. 18, 1927 to T. D. Miller; 1690908 of Nov. 6, 1928 to F. W. Preston; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; **Can. Pats.** 155563 of May 12, 1914 to Calvin Russell; 220781 of Jul. 11, 1922 to Roofing Patents Co.

450 [p. 663] **U. S. Pats.** 430366 of Jun. 17, 1890 to G. H. Babcock (tile); 1059682 of Apr. 22, 1913 to T. D. Miller (metal); 1076123 of Oct. 21, 1913 to O. F. Jacobson (metal); 1145440 of Jul. 6, 1915 to Calvin Russell; 1340348 of May 18, 1920 to C. E. Rahr; 1 89979 of Sep. 6, 1921 to C. E. Rahr and Calvin Russell; 1496336 of Jun. 3, 1924 to M. P. Ennis; 1591042 of Jul. 6, 1926 to W. J. Ingels; 1592014 and 1592015 of Jul. 13, 1926 to J. A. Topping; 1685559 of Sep. 25, 1928 to W. R. Tobias; 1690396 of Nov. 6, 1928 to L. W. Block; 1691343 of Nov. 13, 1928 to J. B. French; 1698032 of Jan. 8, 1929 to G. R. Stark; 1699963 of Jan. 22, 1929 to H. R. French; 1759901 of May 27, 1930 to N. P. Harshberger; 2013556 of Sep. 3, 1935 to C. R. Eckert; 2019701 of Nov. 5, 1935 to M. L. Hamlin; 2028004 of Jan. 14, 1936 to H. T. Nichols; 2047161 of Jul. 7, 1936 to Herbert Abraham; 2073274 of Mar. 9, 1937 to F. C. Young; **Can. Pats.** 166571 of Dec. 14, 1915 to Calvin Russell; 248541 of Apr. 7, 1915 to Flintkote Co.; 268273 of Feb. 8, 1927 to Flintkote Co.; 300414 of May 20, 1930 to Patent & Licensing Corp.; 319769 of Feb. 16, 1932 to H. T. Nichols; 333032 of Jun. 6, 1933 to Barrett Co.; 342751 of Jul. 3, 1934 to Barrett Co.

451 [p. 663] **U. S. Pats.** 1447290 of Mar. 6, 1923 to A. C. Fischer; 1488447 of Mar. 25, 1924 to J. A. Topping; 1552787 of Sep. 8, 1925 to R. M. Weston; 1580096 of Apr. 6, 1926 to F. J. Ballintine; 1592015 of Jul. 13, 1926 to J. A. Topping; 1648692 of Nov. 8, 1927 to J. A. McCarthy; Reissue 17143 of Nov. 20, 1928 to A. C. Fischer; 1717603 of Jun. 18, 1929 to N. P. Harshberger; 1725551 of Aug. 20, 1929 to F. E. Theilacker; Reissue 18698 of Dec. 20, 1932 to N. P. Harshberger.

452 [p. 663] **U. S. Pats.** 595618 of Dec. 14, 1897 to Paul Gambs (slate); 1460795 of Jul. 3, 1923 to N. P. Harshberger (sides folded); 1479035 of Jan. 1, 1924 to A. C. Fischer; 1567538 of Dec. 29, 1925 to H. M. Morgan, Jr.; 1568807 of Jan. 5, 1926 to P. J. Dahm; 1582266 of Apr. 27, 1926 to N. P. Harshberger (sides folded); 1604745 of Oct. 26, 1926 to Dozier Finley; 1637306 of Jul. 26, 1927 to J. E. Hooker; 1641427 of Sep. 6, 1927 to N. P. Harshberger; 1657082 of Jan. 24, 1928 to R. P. Harshberger; 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1658806 of Feb. 14, 1928 to J. W. Kaufer; 1694418 of Dec. 11, 1928 to O. A. Heppes; 1696120 of Dec. 18, 1928 to J. E. Hooker; 1701704 of Feb. 12, 1929 to V. T. Stinson; 1717603 of Jun. 18, 1929 to N. P. Harshberger; 1756742 of Apr. 29, 1930 to N. P. Harshberger and R. P. Harshberger; 1806161 of May 19, 1931 to H. L. Guy; 1820388 of Aug. 25, 1931 to H. R. French; 1828222 of Oct. 20, 1931 to O. B. Clow; 1873944 of Aug. 23, 1932 to J. E. Black; 1891229 of Dec. 20, 1932 to M. L. Hamlin; 1941105 of Dec. 26, 1933 to H. C. Naterman; 1962612 and 1962613 of Jun. 12, 1934 to G. B. Payne; 1974707 of Sep. 25, 1934 to M. A. F. Don; 1983936 of Dec. 11, 1934 to Dozier Finley; 1984529 of Dec. 18, 1934 to N. P. Harshberger; 2004879 of Jun. 11, 1935 to E. C. Matthews and L. M. Clary; 2005335 of Jun. 18, 1935 to R. W. Clough; 2007710 of Jul. 9, 1935 to Dozier Finley; 2018722 of Oct. 19, 1935 to D. B. Humphrey; 2035369 and 2035370 of Mar. 24, 1936 to N. P. Harshberger; 2057245 of Oct. 13, 1936 to A. E. F. Moone; 2076014 of Apr. 6, 1937 to F. H. Bluhm; **Can. Pats.** 185764 of Jul. 30, 1918 to W. H. Garges; 261699 of Jun. 15, 1926 to H. M. Morgan, Jr. (folded); 266411 of Dec. 7, 1926 to P. J. Dahm; 319848 of Feb. 16, 1932 to McHenry-Millhouse Mfg. Co.; 332852 of May 30, 1933 to Barrett Co.; 347155 and 347156 of Jan. 1, 1935 to Barrett Co.; 350917 of Jun. 11, 1935 to Patent & Licensing Corp.; **Brit. Pats.** of 1903 (Feb. 5), 2765 to William Jackson; 121145 of Nov. 23, 1917 to Richard Fletcher and Harry Fletcher.

453 [p. 664] **U. S. Pats.** 1115866 of Nov. 3, 1914 to H. M. Reynolds; 1410867 of Mar. 28, 1922 to Herbert Abraham; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1488447 of Mar. 25, 1924 to J. A. Topping; 1500568 of Jul. 8, 1924 to Herbert Abraham; 1509795 of Sep. 23,

1924 to J. F. Tiner (metal); 1524125 of Jan. 27, 1925 to L. M. Ford; 1544956 of Jul. 7, 1925 to C. T. Torbert; 1642148 of Sep. 13, 1927 to H. L. Guy; 1664285 of Mar. 27, 1928 to R. J. Deans; 1685999 of Oct. 2, 1928 to W. A. Harris; 1729830 of Oct. 1, 1929 to N. P. Harshberger; 1787622 of Jan. 6, 1931 to H. G. Goslin; 1825565 of Sep. 29, 1931 to N. Z. Butterick; 1842761 of Jan. 26, 1932 to J. A. McCarthy; 1902155 of Mar. 21, 1933 to J. E. Berkheimer; 1943953 of Jan. 16, 1934 to J. V. Donahue; **Can. Pats.** 218497 of May 9, 1922 to Herbert Abraham; 233887 of Aug. 28, 1923 to Herbert Abraham; 236137 and 236138 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 333433 of Jun . 20, 1933 to Patent & Licensing Corp.; 358350 of Jun. 9, 1936 to J. A. Topping; **Brit. Pats.** 184361 of Jul. 26, 1921 to Herbert Abraham; 194700 of Mar. 10, 1922 to Herbert Abraham; 202921 of Feb. 27, 1923 to Herbert Abraham.

454 [p. 664] **U. S. Pats.** 1441420 of Jan. 9, 1923 to N. P. Harshberger; 1459827 of Jun. 26, 1923 to N. P. Harshberger; 1472270 of Oct. 30, 1923 to N. P. Harshberger; 1548017 of Aug. 4, 1925 to Herbert Abraham; 1584095 of May 11, 1926 to N. P. Harshberger; 1666203 of Apr. 17, 1928 to N. P. Harshberger; 1849785 of Mar. 15, 1932 to E. R. Black; 2050218 of Aug. 4, 1936 to Herbert Abraham; **Can. Pats.** 218133 of May 2, 1922 to N. P. Harshberger; 357587 of May 5, 1936 to Barrett Co.

455 [p. 664] **U. S. Pats.** 282439 of Jul. 31, 1883 to J. C. Chambers; 387360 of Aug. 7, 1888 to J. H. Keedy; 646623 of Apr. 3, 1900 to S. R. Hawthorne; 1153152 of Sep. 7, 1915 to Francis Brucker; 1270905 of Jul. 2, 1918 to H. W. White; 1322888 of Nov. 25, 1919 to A. C. Fischer; 1345683 of Jul. 6, 1920 to R. R. Mabie; 1356930 of Oct. 26, 1920 to J. R. McCord, Jr.; 1463482 of Jul. 31, 1923 to W. J. Mountford, Jr.; 1468239 of Sep. 18, 1923 to B. C. Kridler; 1477167 of Dec. 11, 1923 to A. C. Fischer; 1493852 of May 13, 1924 to W. A. Fogg; 1498947 of Jun. 24, 1924 to J. O. Bewan; 1510497 of Oct. 7, 1924 to C. L. Keller; 1510756 of Oct. 7, 1924 to L. T. Ayrault and John Ayrault, Jr.; 1521893 of Jan. 6, 1925 to B. C. Kridler and J. C. Boyle; 1537952 of May 19, 1925 to B. C. Kridler and J. C. Boyle; 1544391 of Jun. 30, 1925 to V. J. Harward and W. P. Budd; 1561677 of Nov. 17, 1925 to H. W. White; 1562409 of Nov. 17, 1925 to E. J. Brady; 1575662 of Mar. 9, 1926 to Frank Vandeven; 1579715 of Apr. 6, 1926 to B. C. Kridler and J. C. Boyle; 1582281 of Apr. 27, 1926 to B. C. Kridler and J. C. Boyle; 1613156 of Jan. 4, 1927 to J. C. Bergner; 1651392 of Dec. 6, 1927 to H. H. Honigbaum; 1692388 of Nov. 20, 1928 to W. A. Schollmeyer; 1725466 of Aug. 20, 1929 to P. S. MacMichael; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; 1790860 of Feb. 3, 1931 to A. L. Harvey; **Can. Pats.** 48876 of May 8, 1895 to C. H. Dana; 166322 of Nov. 30, 1915 to Francis Brucker; 243654 of Oct. 14, 1924 to Richardson Co.; 262603 of Jul. 13, 1926 to Beaver Co., Ltd.

456 [p. 664] **U. S. Pats.** 553514 of Jan. 28, 1896 to Frederick Crawford (metal); 1580096 of Apr. 6, 1926 to F. J. Ballintine; 1927436 of Sep. 19, 1933 to A. C. Fischer; **Can. Pat.** 49794 of Aug. 27, 1895 to Frederick Crawford.

457 [p. 664] **U. S. Pats.** 1685999 of Oct. 2, 1928 to W. A. Harris; 1860181 of May 24, 1932 to F. E. Horne; 2059233 of Nov. 3, 1936 to N. P. Harshberger; **Can. Pat.** 264815 of Oct. 5, 1926 to Flintkote Co.

458 [p. 664] **U. S. Pats.** 320979 of Jun. 30, 1885 to L. B. Snow; Design 38931 of Dec. 3, 1907 to J. L. Dickelman; 1100955 of Jun. 23, 1914 to E. B. Coburn; 1104998 of Jul. 28, 1914 to F. C. Overbury; 1198653 of Sep. 19, 1916 to F. C. Overbury; 1301591 of Apr. 22, 1919 to F. C. Overbury; 1322220 of Nov. 18, 1919 to A. L. Baughman; 1547498 of Jul. 28, 1925 to G. M. Jordan; 1575974 of Mar. 9, 1926 to G. E. Conway; 1630019 of May 24, 1927 to F. P. Leonard; 1802868 of Apr. 28, 1931 to E. R. Black; 1819717 of Aug. 18, 1931 to C. L. Mangano; 1885346 of Nov. 1, 1932 to N. P. Harshberger; 1980760 of Nov. 13, 1934 to T. M. Mayfield; 2031993 of Feb. 25, 1936 to P. S. Varden; **Can. Pats.** 265985 of Nov. 23, 1926 to G. E. Conway; 315466 of Sep. 22, 1931 to Johns-Manville Corp.; 316830 of Nov. 3, 1931 to C. L. Mangano and R. M. Fleming.

459 [p. 665] **U. S. Pats.** 1076123 of Oct. 21, 1913 to O. F. Jacobson (metal); 1496336 of Jun. 3, 1924 to M. P. Ennis; 1582266 of Apr. 27, 1926 to N. P. Harshberger; 1667185 of Apr. 24, 1928 to W. E. Bartels; 1698032 of Jan. 8, 1929 to G. R. Stark; 1885346 of Nov. 1, 1932 to N. P. Harshberger; 2017230 of Oct. 15, 1935 to E. R. Black.

460 [p. 665] **U. S. Pats.** 1385809 of Jul. 26, 1921 to Herbert Abraham; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; **Can. Pat.** 214368 of Nov. 22, 1921 to Herbert Abraham (Ruberoid Co., Ltd.); **Brit. Pat.** 180150 of May 25, 1922 to Herbert Abraham.

461 [p. 665] **U. S. Pats.** 1438571 of Dec. 12, 1922 to Herbert Abraham; 1491015 of Apr. 22, 1924 to O. D. McFarland; 1604708 of Oct. 26, 1926 to G. W. Mills, Jr.; **Can. Pats.** 268272 and 268273 of Feb. 8, 1927 to Flintkote Co.; **Brit. Pat.** 219718 of Feb. 27, 1923 to Herbert Abraham.

462 [p. 665] **U. S. Pat.** 1119439 of Dec. 1, 1924 to R. B. Marschke; 2054917 of Sep. 22, 1936 to E. S. Yunik; **Can. Pat.** 266501 of Dec. 7, 1926 to Barrett Co.

463 [p. 665] **U. S. Pats.** 838232 of Dec. 11, 1906 to J. O. Ballentine; 1512248 of Oct. 21, 1924 to James Smith and M. E. Constable; 1513940 of Nov. 4, 1924 to James Smith and M. E. Constable; 1584054 of May 11, 1926 to H. R. Wardell; 1645767 of Oct. 18, 1927 to T. D. Miller; 1699213 of Jan. 15, 1929 to C. A. Statler; 1754771 of Apr. 15, 1930 to Lester Kirschbraun; 1759901 of May 27, 1930 to N. P. Harshberger; **Can. Pats.** 245173 of Dec. 9, 1924 to Johns-Manville Co.; 302453 of Jul. 29, 1930 to L. F. Lindley; 357757 of May 12, 1936 to Barrett Co.

464 [p. 665] **U. S. Pats.** 1201811 of Oct. 17, 1916 to H. H. Dupont; 1666088 of Apr. 17, 1928 to J. W. Farr; 1702414 and 1702415 of Feb. 19, 1929 to C. J. Richards; 1865771 of Jul. 5, 1932 to Solomon Levy; 1983936 of Dec. 11, 1934 to Dozier Finley; **Ger. Pat.** Design 331693 of Jan. 2, 1908 to Fr. Sieges & Sohn.

465 [p. 665] **U. S. Pat.** 1792543 of Feb. 17, 1931 to G. E. Livingston and W. H. C. Ness.

466 [p. 665] **U. S. Pat.** 1943258 of Jan. 9, 1934 to N. P. Harshberger; 2037297 of Apr. 14, 1936 to F. W. Yeager; 2065478 of Dec. 22, 1936 to Joseph Schulman.

467 [p. 665] **U. S. Pats.** 115912 of Jun. 13, 1871 to C. G. Van Pappelendam (metal); 117601 of Aug. 1, 1871 to C. W. Chaffee; 154828 of Sep. 8, 1874 to Edwin Bennett (tile); 170582 of Nov. 30, 1875 to C. J. Merrill (tile); 211955 of Feb. 4, 1879 to Edwin Bennett (tile); 267904 of Nov. 21, 1882 to Lorenzo Lane and L. D. Woodworth (tile); Design 18208 of Mar. 27, 1888 to J. S. Thorn (metal); Design 20402 o Dec. 16, 1890 to G. H. Babcock (metal); 430365, 430369 and 430371 of Jun. 17, 1890 to G. H. Babcock (tile); Design 20635 of Mar. 24, 1891 to G. H. Babcock (metal); 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 595618 of Dec. 14, 1897 to Paul Gambs (slate); 605654 of Jun. 14, 1898 to Gustav Schulze (tile); Design 39274 of Apr. 21, 1908 to S. A. Jones (tile); 888825 of May 26, 1908 to H. E. Kock; 1070738 of Aug. 19, 1913 to Calvin Russell; 1256508 of Feb. 19, 1918 to M. B. Becker; 1690908 of Nov. 6, 1928 to F. W. Preston; 1772488 of Aug. 12, 1930 to B. E. Horne; 1799293 of Apr. 7, 1931 to H. R. French; 1928285 of Sep. 26, 1933 to A. C. Fischer; **Can. Pats.** 44903 of Dec. 18, 1893 to J. M. Olsen, H. P. Olsen and F. W. Bronn (tile); 306998 of Dec. 23, 1930 to Patent & Licensing Corp.; **Ger. Pats.** 4940 of Aug. 2, 1878 to Hartwig Hüser; 6174 of Jul. 21, 1878 to Alex Brink (slate); **Norwegian Pat.** 9016 of Jul. 11, 1900 to C. J. Walls (cement).

468 [p. 665] **U. S. Pats.** 207989 of Sep. 10, 1878 to H. W. Shepard (metal); 267674 of Nov. 21, 1882 to Chester Comstock (metal); 292585 of Jan. 29, 1884 to E. B. Repp (metal); 294256 of Feb. 26, 1884 to L. H. Montross and J. C. West (metal); 309459 of Dec. 16, 1884 to M. F. Hamsley (metal); 341188 of May 4, 1886 to Ezekiel Van Noorden (metal); 482025 of Sep. 6, 1892 to E. B. Repp (metal); 562798 of Jun. 30, 1896 to Heinrich Bröcker (tile); 1515749 of Nov. 18, 1924 to N. G. Olsson (metal); 1566415 of Dec. 22, 1925 to Robert Miller (metal); 1637306 of Jul. 26, 1927 to J. E. Hooker; 1641427 of Sep. 6, 1927 to N. P. Harshberger; 1688612 of Oct. 23, 1928 to O. B. Clow; 1694418 of Dec. 11, 1928 to O. A. Heppes; 1696120 of Dec. 18, 1928 to J. E. Hooker; 1877222 of Sep. 13, 1932 to F. R. Brydle; 1968228 of Jul. 31, 1934 to Wright Smith, Jr.; 1983936 of Dec. 11, 1934 to Dozier Finley; 2068756 of Jan. 26, 1937 to S. P. Miller; **Can. Pats.** 260007 of Apr. 20, 1926 to Flintkote Co.; 301311 of Jun. 17, 1930 to J. E. Hooker.

469 [p. 665] **U. S. Pats.** 5091 of May 1, 1847 to Mathew Stewart (metal); 241805 of May 24, 1881 to W. G. Hyndman (metal); 790127 of May 16, 1905 to Ludwig Hatschek (tile); 1359167 of Nov. 16, 1920 to A. A. Griswold; 1430338 of Sep. 26, 1922 to J. A. Topping Reissue 15556 of Mar. 6, 1923 to J. A. Topping; 1492516 of Apr. 29, 1924 to C. L. Keller 1493374 of May 6, 1924 to W. J. Mountford, Jr.; 1496912 of Jun. 10, 1924 to J. A. Topping

1508581 of Sep. 16, 1924 to John Shea; 1538235 of May 19, 1925 to A. L. Bell and F. A. Browne; 1552708 of Sep. 8, 1925 to W. A. Joy; 1557616 of Oct. 20, 1925 to W. J. Riley; 1561677 of Nov. 17, 1925 to H. W. White; 1566415 of Dec. 22, 1925 to Robert Miller; 1574345 of Feb. 23, 1926 to C. C. Gates; 1576423 of Mar. 9, 1926 to Jesse Fulenwider and Harry Fulenwider; 1582018 of Apr. 20, 1926 to J. C. Bergner; 1593407 and 1593408 of Jul. 20, 1926 to H. H. Honigbaum; 1618077 of Feb. 15, 1927 to Harry Fulenwider and Jesse Fulenwider; 1626780 of May 3, 1927 to W. G. Bickell; 1678804 of Jul. 31, 1928 to C. F. Ames, Jr.; 1681765 of Aug. 21, 1928 to C. C. Figge; 1686675 of Oct. 9, 1928 to C. F. Ames, Jr.; 1722962 of Jul. 30, 1929 to O. A. Heppes; 1730653 of Oct. 8, 1929 to G. G. Guertin; 1772487 and 1772488 of Aug. 12, 1930 to B. E. Horne; 1813798 of Jul. 7, 1931 to Anthony Gerosa; 1817743 of Aug. 4, 1931 to C. C. Figge; 1978841 of Oct. 30, 1934 to G. A. Holman; 2007855 of Jul. 9, 1935 to H. L. Guy; **Can. Pats.** 48037 of Jan. 28, 1895 to E. B. Repp (metal); 213089 of Aug. 23, 1921 to A. A. Griswold; 240231 of May 20, 1924 to J. A. Topping; 260006 of Apr. 20, 1926 to Flintkote Co.; **French Pat.** 455421 of Mar. 11, 1913 to Louis Bordat (cement).

470 [p. 665] **U. S. Pats.** 158123 of Dec. 22, 1874 to Joseph Ravoux (metal); 262475 of Aug. 8, 1882 to E. B. Repp (metal); 1436945 of Nov. 28, 1922 to R. K. Clifton; 1462772 of Jul. 24, 1923 to E. R. Snyder and C. W. Mortimer; 1666046 of Apr. 10, 1928 to O. A. Heppes; **Can. Pats.** 248798 of Apr. 21, 1925 to H. M. McLaughlin (metal); 260008 of Apr. 20, 1926 to Flintkote Co.

471 [p. 665] **U. S. Pats.** 1678333 of Jul. 24, 1928 to C. C. Figge; 1819717 of Aug. 18, 1931 to C. L. Mangano; 1820015 of Aug. 25, 1931 to C. C. Figge; 2017230 of Oct. 15, 1935 to E. R. Black; **Can. Pat.** 26965 of Jun. 16, 1887 to Nelson Green (metal).

472 [p. 666] **U. S. Pats.** 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 316134 of Apr. 21, 1885 to M. G. Farmer (metal); 341967 of May 18, 1886 to J. S. Thorn and Charles Leffler (metal); Design 18209 of Mar. 27, 1888 to J. S. Thorn (metal); Design 19139 of Jun. 4, 1889 to L. L. Sagendorph (metal); 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 516570 of Mar. 13, 1894 to Nicholas Monshausen (metal); 691650 of Jan. 21, 1902 to Nicholas Monshausen (metal); 1108236 of Aug. 25, 1914 to H. M. Reynolds; 1274605 of Aug. 6, 1918 to Calvin Russell; Design 56106 of Aug. 17, 1920 to Calvin Russell; 1596449 of Aug. 17, 1926 to C. E. Rahr; 1604745 of Oct. 26, 1926 to Dozier Finley; 1675533 of Jul. 3, 1928 to H. A. Cumfer; 1679883 of Aug. 7, 1928 to A. S. Speer; 1690908 of Nov. 6, 1928 to F. W. Preston; 1691343 of Nov. 13, 1928 to J. B. French; 1699963 of Jan. 22, 1929 to H. R. French; 1744490 of Jan. 21, 1930 to C. W. Mortimer; 1745631 of Feb. 4, 1930 to A. O. Mickelson; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; 1775927 of Sep. 16, 1930 to Walter Becker; 1799293 of Apr. 7, 1931 to H. R. French; 1848965 of Mar. 8, 1932 to T. D. Miller; 1849779 of Mar. 15, 1932 to Harry Zimmerman; 1961896 of Jun. 5, 1934 to A. L. Barrall; **Can. Pats.** 23451 of Feb. 18, 1886 to J. B. Blaikie (slate); 260668 of May 11, 1926 to Barrett Co.; 285715 of Dec. 18, 1928 to Flintkote Co.; 286001 of Jan. 1, 1929 to J. B. French; 306998 of Dec. 23, 1930 to Patent & Licensing Corp.; 364890 of Mar. 16, 1937 to J. A. Topping; **Brit. Pat.** of 1867 (Mar. 17), 2612 to William Le Duc (slate); **Ger. Pats.** 182317 of Mar. 9, 1907 to H. L. Schwab (cement); 249986 of Aug. 30, 1910 to Naumann Schefftel.

473 [p. 666] **U. S. Pats.** 124963 of Mar. 26, 1872 to Charles Lewando (metal); 207989 of Sep. 10, 1878 to H. W. Shepard (metal); 267674 of Nov. 21, 1882 to Chester Comstock (metal); 292585 of Jan. 29, 1884 to E. B. Repp (metal); 294256 of Feb. 26, 1884 to L. H Montross and J. C. West (metal); 1431102 of Oct. 3, 1922 to J. F. Donahue and M. G. Staley; 1431103 of Oct. 3, 1922 to J. F. Donahue; 1469041 of Sep. 25, 1923 to H. L. Guy; 1486970 of Mar. 18, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1510534 and 1510535 of Oct. 7, 1924 to Herbert Abraham; 1524125 of Jan. 27, 1925 to L. M. Ford; 1558795 of Oct. 27, 1925 to J. F. Donahue; 1567817 of Dec. 29, 1925 to J. A. Scharwath; 1576090 of Mar. 9, 1926 to S. E. Carpenter; 1601735 of Oct. 5, 1926 to W. A. Harris; 1612700 of Dec. 28, 1926 to J. V. Cook; 1613099, 1613100 and 1613103 of Jan. 4, 1927 to N. P. Harshberger; 1648692 of Nov. 8, 1927 to J. A. McCarthy; 1658806 of Feb. 14, 1928 to J. W. Kaufer; 1663658 of Mar. 27, 1928 to N. P. Harshberger; 1669723 of May 15, 1928 to H. T. Nichols; 1669981 of May 15, 1928 to W. A. Harris; 1672713 of Jun. 5, 1928 to W. R. Durbin; 1674008 of Jun. 19, 1928 to H. L. Guy; 1688612 of Oct. 23, 1928 to O. B. Clow; 1696120 of Dec. 18, 1928 to J. E. Hooker;

1710954 of Apr. 30, 1929 to F. E. Theilacker; 1729830 of Oct. 1, 1929 to N. P. Harshberger; 1732229 of Oct. 22, 1929 to W. A. Harris; 1762404 and 1762405 of Jun. 10, 1930 to A. O. Mickelson and D. A. Cumfer; 1772487 of Aug. 12, 1930 to B. E. Horne; Reissue 17862 of Nov. 11, 1930 to W. A. Harris; Reissue 18515 of Jul. 12, 1932 to F. E. Theilacker; 1877222 of Sep. 13, 1932 to F. R. Brydle; 1889121 of Nov. 29, 1932 to N. P. Harshberger; 1893028 of Jan. 3, 1933 to H. H. Honigbaum; 1901161 of Mar. 14, 1933 to N. P. Harshberger; 1925239 of Sep. 5, 1933 to Harry Forbes; 1926070 of Sep. 12, 1933 to D. H. Sweet; 1934831 of Nov. 14, 1933 to D. H. Sweet; Reissue 19637 of Jul. 9, 1935 to N. P. Harshberger; 2013002 of Sep. 3, 1935 to G. S. Logan; 2055758 of Sep. 29, 1936 to J. A. Topping; **Can. Pats.** 229746 of Mar. 27, 1923 to J. F. Donahue and M. G. Staley; 229747 of Mar. 27, 1923 to J. F. Donahue; 236342 of Dec. 11, 1923 to H. L. Guy; 238434 of Mar. 11, 1924 to J. F. Donahue; 249097 of Apr. 28, 1925 to O. A. Heppes; 253786 of Sep. 22, 1925 to A. O. Mickelson; and D. A. Cumfer; 255977 of Dec. 1, 1925 to Ford Roofing Prods. Co.; 259447 of Mar. 30, 1926 to H. L. Guy and C. S. Purnell; 263976 of Aug. 31, 1926 to Flintkote Co.; 267703 of Jan. 18, 1927 to Flintkote Co.; 269330 of Mar. 29, 1927 to N. P. Harshberger; 279378 of Apr. 17, 1928 to N. P. Harshberger; 283989 of Oct. 16, 1928 to H. H. Honigbaum; 301311 of Jun. 17, 1930 to J. E. Hooker; 309864 of Mar. 31, 1931 to Barrett Co.; 312125 of Jun. 9, 1931 to Barrett Co.; 342750 of Jul. 3, 1934 to Barrett Co.; 352314 of Aug. 13, 1935 to Black Systems, Inc.; 354389 of Nov. 26, 1935 to Barrett Co.; 363191 of Jan. 5, 1937 to Lehon Co.

474 [p. 666] **U. S. Pats.** 1520947 of Dec. 30, 1924 to C. H. Harris; 1532421 of Apr. 7, 1925 to Hugh MacInnes and W. J. Riley; 1540960 of Jun. 9, 1925 to Roy Sherman; 1547469 of Jul. 28, 1925 to J. A. Topping; 1556649 of Oct. 13, 1925 to J. A. Topping; 1557392 of Oct. 13, 1925 to Herbert Abraham; 1557616 of Oct. 20, 1925 to W. J. Riley; 1560276 of Nov. 3, 1925 to Hugh MacInnes; 1570222 of Jan. 19, 1926 to J. C. Barley; 1582727 of Apr. 27, 1926 to J. J. Bradfield; 1612718 of Dec. 28, 1926 to J. F. Grice; 1626780 of May 3, 1927 to W. G. Bickell; 1659903 of Feb. 21, 1928 to P. C. Wolf; 1668291 of May 1, 1928 to J. A. Topping; 1722962 of Jul. 30, 1929 to O. A. Heppes; 1730347 of Oct. 8, 1929 to C. F. Ames, Jr. and C. W Mortimer; 1748327 of Feb. 25, 1930 to E. R. Black; 1750331 and 1750332 of Mar. 11,1930. to W. A. Schollmeyer; 1753583 of Apr. 8, 1930 to A. S. Speer; 1873944 of Aug. 23, 1932 to J. E. Black; 1962197 of Jun. 12, 1934 to H. H. Honigbaum; 2007855 of Jul. 9, 1935 to H. L. Guy; 2041780 of May 26, 1936 to W. L. Rowe; **Can. Pats.** 260006 of Apr. 20, 1926 to Flintkote Co.; 266290 of Nov. 30, 1926 to Beaver Co., Ltd.; 286851 of Jan. 29, 1929 to Lehon Co.; **French Pat.** 402181 of Sep. 30, 1909 to Carl Streckfuss (cement).

475 [p. 666] **U. S. Pats.** 124963 of Mar. 26, 1872 to Charles Lewando (metal); 140928 of Jul. 15, 1873 to Charles Lewando (metal); 158123 of Dec. 22, 1874 to Joseph Ravoux; 262475 of Aug. 8, 1882 to E. B. Repp (metal); 320979 of Jun. 30, 1885 to L. B. Snow; 326374 of Sep. 15, 1885 to B. B. Adams; 406024 of Jul. 2, 1889 to J. D. Burton (metal); 740842 of Oct. 6, 1903 to Albert Friedley (metal); 1186619 of Jun. 13, 1916 to L. F. Tiefel and J. B. Tiefel (metal); 1427154 of Aug. 29, 1922 to A. A. Griswold; 1431476 of Oct. 10, 1922 to Hugh MacInnes; 1434116 of Oct. 31, 1922 to A. J. Hauber; 1436813 of Nov. 28, 1922 to Hugh MacInnes; 1436945 of Nov. 28, 1922 to R. K. Clifton; 1483882 of Feb. 19, 1924 to W. F. Harvey; 1508789 of Sep. 16, 1924 to C. H. Harris; 1529530 of Mar. 10, 1925 to R. L. Wyatt; 1533923 of Apr. 14, 1925 to R. A. Knoll; 1539716 of May 26, 1925 to W. T. Conley; 1570516 of Jan. 19, 1926 to T. D. Miller; 1588304 of Jun. 8, 1926 to A. L. Brownrigg; 1589719 of Jun. 22, 1926 to George Ritter; 1601735 of Oct. 5, 1926 to W. A. Harris; 1614871 of Jan. 18, 1927 to F. R. Brydle; 1625308 of Apr. 19, 1927 to William Freegard; 1630019 of May 24, 1927 to F. P. Leonard; 1634972 of Jul. 5, 1927 to N. R. Bartlett; 1635733 of Jul. 12, 1927 to G. R. Wyman; 1636349 of Jul. 19, 1927 to R. B. Adams; 1640806 of Aug. 30, 1927 to George Ritter; 1659575 of Feb. 21, 1928 to George Ritter; 1666046 of Apr. 10, 1928 to O. A. Heppes; 1669981 of May 15, 1928 to W. A. Harris; 1671424 of May 29, 1928 to N. P. Harshberger; 1697519 of Jan. 1, 1929 to W. W. Weaver; 1731187 of Oct. 8, 1929 to R. B. Adams; 1732229 of Oct. 22, 1929 to W. A. Harris; 1762404 and 1762405 of Jun. 10, 1930 to A. O. Mickelson and D. A. Cumfer; 1772487 of Aug. 12, 1930 to B. E. Horne; 1774861 of Sep. 2, 1930 to J. L. Wettlaufer; 1782416 of Nov. 25, 1930 to William Freegard; 1927436 of Sep. 19, 1933 to A. C. Fischer; **Can. Pats.** 220118, 220119 and 220120 of Jun. 27, 1922 to A. A. Griswold and J. F. Chevalier;

241178 of Jul. 1, 1924 to W. F. Harvey; 260008 of Apr. 20, 1926 to Flintkote Co.; 263976 of Aug. 31, 1926 to Flintkote Co.; 267703 and 267704 of Jan. 18, 1927 to Flintkote Co.; 272312 of Jul. 12, 1927 to Building Products Co., Ltd.; 278448 of Mar. 13, 1928 to R. H. Adams; 302350 of Jul. 22, 1930 to Patent & Licensing Corp.; 356256 of Mar. 3, 1936 to Bakelite Building Products Co., Inc.

476 [p. 666] **U. S. Pats.** 326374 of Sep. 15, 1885 to B. B. Adams; 1274410 of Aug. 6, 1918 to A. A. Griswold; 1279684 of Sep. 24, 1918 to A. A. Griswold; 1320136 of Oct. 28, 1919 to A. A. Griswold; 1321623 and 1321624 of Nov. 11, 1919 to A. A. Griswold; 1359167 of Nov. 16, 1920 to A. A. Griswold; 1427732 of Aug. 29, 1922 to A. A. Griswold; 1447019 of Feb. 27, 1923 to A. A. Griswold; 1450182 of Apr. 3, 1923 to Anthony Lamm; 1470837 of Oct. 16, 1923 to W. J. Hofstatter; 1483046 of Feb. 5, 1924 to George Ritter; 1483882 of Feb. 19, 1924 to W. F. Harvey; 1484020 of Feb. 19, 1924 to A. A. Griswold; 1498356 of Jun. 17, 1924 to Isadore Davis; 1508789 of Sep. 16, 1924 to C. H. Harris; 1512400 of Oct. 21, 1924 to F. R. Brydle; 1513800 of Nov. 4, 1924 to F. R. Brydle; 1522234 of Jan. 6, 1925 to William Freegard and M. S. Darrow; 1524432 of Jan. 27, 1925 to A. A. Griswold; 1548367 of Aug. 4, 1925 to F. E. Lietz; 1568750 of Jan. 5, 1926 to F. E. Lietz; 1571582 of Feb. 2, 1926 to A. A. Griswold and A. C. Goddard; 1582804 of Apr. 27, 1926 to J. W. Towler; 1583254 of May 4, 1926 to F. E. Lietz; 1589719 of Jun. 22, 1926 to George Ritter; 1593551 of Jul. 20, 1926 to Charles O'Dell; 1607455 of Nov. 16, 1926 to A. A. Griswold and A. C. Goddard; 1608738 of Nov. 30, 1926 to H. P. Hayden and W. W. Weaver; 1623127 of Apr. 5, 1927 to A. E. F. Moone; 1623474 of Apr. 5, 1927 to W. H. Hale; 1629558 of May 24, 1927 to H. R. Wardell; 1646551 of Oct. 25, 1927 to A. E. F. Moone; Reissue 16832 of Dec. 27, 1927 to Charles O'Dell; 1661067 of Feb. 28, 1928 to Harry Fulenwider and Jesse Fulenwider; 1666939 of Apr. 24, 1928 to J. F. Lietz; 1678804 of Jul. 31, 1928 to C. F. Ames, Jr.; 1684479 of Sep. 18, 1928 to C. R. Eckert; Reissue 17133 of Nov. 13, 1928 to F. E. Lietz; 1703156 of Feb. 26, 1929 to F. E. Lietz; 1744656 of Jan. 21, 1930 to C. J. McDavitt; 1757351 of May 6, 1930 to F. W. Yeager and W. P. Schulz; 1765058 of Jun. 17, 1930 to M. S. Darrow, W. J. Hart, F. G. Gronemeyer and F. B. Watkins; 1772924 of Aug. 12, 1930 to J. H. Weller; Reissue 17957 of Feb. 10, 1931 to J. F. Lietz; 1802868 of Apr. 28, 1931 to E. R. Black; 1834966 of Dec. 8, 1931 to F. C. Overbury; 1842564 of Jan. 26, 1932 to F. C. Overbury; 1850088 of Mar. 22, 1932 to F. L. O. Wadsworth; 1895038 of Jan. 24, 1933 to J. O. Jones; 1927696 of Sep. 19, 1933 to Addison Applegate; 1940936 of Dec. 26, 1933 to E. R. Black; 1959519 of May 22, 1934 to E. R. Black; 1980760 of Nov 13, 1934 to T. M. Mayfield; **Can. Pats.** 24703 of Aug. 10, 1886 to B. B. Adams; 195071 of Dec. 16, 1919 to A. A. Griswold and J. F. Chevalier; 202333 of Jul. 27, 1920 to A. A. Griswold and J. F. Chevalier; 213089 of Aug. 23, 1921 to A. A. Griswold and J. F. Chevalier; 220117 of Jun. 27, 1922 to A. A. Griswold and J. F. Chevalier; 229746 and 229747 of Mar. 27, 1923 to J. F. Donahue; 231125 of May 15, 1923 to A. A. Griswold; 240310 of May 20, 1924 to C. H. Harris; 240467 of May 27, 1924 to Latite Shingle Corp.; 258942 of Mar. 16, 1926 to F. E. Lietz; 271758 of Jun. 21, 1927 to Barrett Co.; 273843 of Sep. 13, 1927 to Barber Asphalt Co.; 282018 of Jul. 24, 1928 to F. E. Lietz and J. F. Lietz; 310684 of Apr. 21, 1931 to Lehon Co.; **Brit. Pats.** 144699 of Apr. 25, 1918 to A. A. Griswold; 174268 of Jan. 26, 1922 to A. A. Griswold and J. F. Chevalier; **Ger. Pat.** 41774 of Mar. 31, 1887 to W. T. Westen.

477 [p. 666] **U. S. Pat.** 2008575 of Jul. 16, 1935 to Ernest Bussey; **Can. Pat.** 359770 of Aug. 11, 1936 to Starite Co.

478 [p. 666] **U. S. Pats.** Design 1467 of Aug. 13, 1861 to Daniel Rickerby (slate); 121063 of Nov. 21, 1871 to O. J. Pierce (metal); 141215 of Jul. 29, 1873 to W. J. Fryer, Jr. and G. H. Johnson (slate); Design 19139 of Jun. 4, 1889 to L. L. Sagendorph (metal); Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; **Ger. Pat.** 22587 of Jun. 2, 1883 to Robert Spahlmann (title).

479 [p. 666] **U. S. Pats.** 1450731 of Apr. 3, 1923 to R. S. Maclean; 1510534 of Oct. 7, 1924 to Herbert Abraham; 1576090 of Mar. 9, 1926 to S. E. Carpenter; 1595080 of Aug. 10, 1926 to A. C. Fischer; 1612700 of Dec. 28, 1926 to J. V. Cook; 1920474 of Aug. 1, 1933 to A. F. Martin; 1934666 of Nov. 7, 1933 to H. G. Goslin; Design 95242 of Apr. 16, 1935 to H. G. Goslin.

480 [p. 666] **U. S. Pat.** 1987133 of Jan. 8, 1935 to M. C. Smith.

481 [p. 666] **U. S. Pats.** Design 19888 of Jun. 10, 1890 to G. H. Babcock (metal); 1795277 of Mar. 3, 1931 to A. C. Fischer.

482 [p. 666] **U. S. Pat.** 320979 of Jun. 30, 1885 to L. B. Snow.

483 [p. 666] **U. S. Pats.** 117601 of Aug. 1, 1871 to C. W. Chaffee; 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 310192 of Jan. 6, 1885 to J. T. Edson; 316134 of Apr. 21, 1885 to M. G. Farmer (metal); 430362 and 430364 of Jun. 17, 1890 to G. H. Babcock (metal); Design 24084 of Mar. 5, 1895 to W. H. Mullins (metal); 888825 of May 26, 1908 to H. E. Kock; 954019 of Apr. 5, 1910 to O. O. Burnett (cement); 1256508 of Feb. 19, 1918 to M. B. Becker; 1799293 of Apr. 7, 1931 to H. R. French; **Can. Pat.** 306998 of Dec. 23, 1930 to Patent & Licensing Corp.

484 [p. 666] **U. S. Pats.** 1441420 of Jan. 9, 1923 to N. P. Harshberger; 1604745 of Oct. 26, 1926 to Dozier Finley; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1729830 of Oct. 1, 1929 to N. P. Harshberger; Reissue 18772 of Mar. 21, 1933 to N. P. Harshberger; 2059234 of Nov. 3, 1936 to N. P. Harshberger; **Can. Pat.** 340686 of Apr. 10, 1934 to E. J. Hetzel.

485 [p. 666] **U. S. Pat.** 1584739 of May 18, 1926 to Isadore Davis.

486 [p. 666] **U. S. Pat.** Design 77218 of Dec. 18, 1928 to A. J. Caron.

487 [p. 666] **U. S. Pats.** 320979 of Jun. 30, 1885 to L. B. Snow; 1737977 of Dec. 3, 1929 to George Ritter.

488 [p. 668] **U. S. Pats.** 1492609 and 1492610 of May 6, 1924 to J. T. Simpson; 1550693 of Aug. 25, 1925 to H. G. Goslin; 1596680 of Aug. 17, 1926 to W. E. Nelson; 1629287 of May 17, 1927 to S. K. Milligan; 1657271 of Jan. 24, 1928 to W. E. Nelson; 1701640 of Feb. 12, 1929 to F. E. Sherriff; Design 91744 of Mar. 13, 1934 to J. A. Topping; Design 93642 of Oct. 16, 1934 to J. A. Topping.

489 [p. 668] **U. S. Pat.** 1783839 of Dec. 2, 1930 to Henri Gauvin; **Can. Pat.** 306211 of Dec. 2, 1930 to Henri Gauvin.

490 [p. 668] **U. S. Pats.** 1320502 of Nov. 4, 1919 to J. T. Simpson; 1492609 and 1492610 of May 6, 1924 to J. T. Simpson.

491 [p. 668] **U. S. Pats.** 1070738 of Aug. 19, 1913 to Calvin Russell; 1116149 of Nov. 3, 1914 to A. S. Spiegel; Reissue 14058 of Feb. 8, 1916 to Calvin Russell; 1274605 of Aug. 6, 1918 to Calvin Russell.

492 [p. 668] **U. S. Pats.** 1690908 of Nov. 6, 1928 to F. W. Preston; 1699963 of Jan. 22, 1929 to H. R. French; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; **Can. Pat.** 285715 of Dec. 18, 1928 to Flintkote Co.; **Ger. Pat.** 249986 of Aug. 30, 1910 to Naumann Schefftel.

493 [p. 668] **U. S. Pats.** Design 1467 of Aug. 13, 1861 to Daniel Rickerby (metal); 141215 of Jul. 29, 1873 to W. J. Fryer, Jr. and G. H. Johnson (slate); 1787622 of Jan. 6, 1931 to H. G. Goslin.

494 [p. 668] **U. S. Pats.** 117601 of Aug. 1, 1871 to C. W. Chaffee; 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 310192 of Jan. 6, 1885 to J. T. Edson; 316134 of Apr. 21, 1885 to M. G. Farmer (metal); 888825 of May 26, 1908 to H. E. Kock.

495 [p. 668] **U. S. Pat.** 1690908 of Nov. 6, 1928 to F. W. Preston.

496 [p. 668] **U. S. Pat.** 954019 of Apr. 5, 1910 to O. O. Burnett (cement); **Can. Pat.** 354135 of Nov. 12, 1935 to United Shoe Machinery Corp.

497 [p. 668] **U. S. Pats.** 309433 of Dec. 16, 1884 to Charles Baillie; 414427 of Nov. 5, 1889 to C. A. Miller; 512986 of Jan. 16, 1894 to L. H. Montross; 908125 of Dec. 29, 1908 to F. C. Overbury; 1077095 of Oct. 28, 1913 to W. S. Orth; 1107762 of Aug. 18, 1914 to H. A. Cumfer; 1110238 of Sep. 8, 1914 to A. S. Spiegel; 1119439 of Dec. 1, 1914 to R. B. Marschke; 1150298 of Aug. 17, 1915 to F. C. Overbury; 1182416 and 1182417 of May 9, 1916 to F. C. Overbury; 1191297 of Jul. 18, 1916 to P. G. Gardner; 1192132 of Jul. 25, 1916 to A. S. Spiegel; 1249544 of Dec. 11, 1917 to A. S. Spiegel; 1257321 of Feb. 26, 1918 to H. A. Cumfer and O. D. McFarland; 1260088 of Mar. 19, 1918 to A. S. Spiegel; 1261280 of Apr. 2, 1918 to F. C. Overbury; 1263987 of Apr. 23, 1918 to G. R. Wyman; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1301332 of Apr. 22, 1919 to A. S. Spiegel; 1301964 of Apr. 29, 1919 to F. C. Overbury; 1310082 of Jul. 15, 1919 to H. G. Hose; 1314476 and 1314477

of Aug. 26, 1919 to F. C. Overbury; 1340402 of May 18, 1920 to F. C. Overbury; 1358113 of Nov. 9, 1920 to H. H. Robertson; 1394342 of Oct. 18, 1921 to F. C. Overbury; 1394911 of Oct. 25, 1921 to R. D. King; 1410018 of Mar. 21, 1922 to O. P. Kiracofe; Reissue 15328 of Apr. 4, 1922 to F. C. Overbury; 1431869 of Oct. 10, 1922 to A. R. Bergen; 1441359 of Jan. 9, 1923 to S. M. Langston; 1441708 of Jan. 9, 1923 to F. C. Overbury; 1455342 of May 15, 1923 to S. M. Langston; 1462138 of Jul. 17, 1923 to S. M. Langston; 1464309 of Aug. 7, 1923 to H. A. Cumfer; 1473377 of Nov. 6, 1923 to S. M. Langston; 1478998 of Jan. 1, 1924 to W. R. Howard and W. H. Gregg; 1487661, 1487662 and 1487663 of Mar. 18, 1924 to S. M. Langston; 1489135 of Apr. 1, 1924 to S. M. Langston; 1489890 of Apr. 8, 1924 to S. M. Langston; 1501162 of Jul. 15, 1924 to H. A. Cumfer; 1515530 of Nov. 11, 1924 to P. P. Welty; 1532538 of Apr. 7, 1925 to S. M. Langston; 1551052 of Aug. 25, 1925 to R. T. Pollock; 1563245 of Nov. 24, 1925 to P. P. Welty; 1567920 of Dec. 29, 1925 to H. A. Cumfer; 1581236 of Apr. 20, 1926 to A. S. Speer; 1593594 of Jul. 27, 1926 to R. P. Perry; 1596449 of Aug. 17, 1926 to C. E. Rahr; 1599512 of Sep. 14, 1926 to W. H. Cady; 1601128 of Sep. 28, 1926 to O. D. McFarland; 1601731 of Oct. 5, 1926 to John Flood; 1612776 of Dec. 28, 1926 to Lester Kirschbraun; 1627665 of May 10, 1927 to F. C. Overbury; 1665450 of Apr. 10, 1928 to H. A. Garber; 1669999 of May 15, 1928 to C. M. Olsen; 1687873 of Oct. 16, 1928 to O. D. McFarland; Reissue 17187 of Jan. 8, 1929 to R. P. Perry; 1745631 of Feb. 4, 1930 to A. O. Mickelson; 1751945 of Mar. 25, 1930 to F. C. Overbury; 1759901 of May 27, 1930 to N. P. Harshberger; 1762330 of Jun. 10, 1930 to H. R. Fielder and Amandus Grau; 1777076 of Sep. 30, 1930 to H. A. Cumfer; 1779916 of Oct. 28, 1930 to R. F. Paup; 1788122 of Jan. 6, 1931 to E. S. Petersen; 1816329 of Jul. 28, 1931 to R. T. Johnston; 1831422 of Nov. 10, 1931 to E. C. Otis; 1834004 of Dec. 1, 1931 to F. C. Overbury and O. A. Heppes; 1973863 of Sep. 18, 1934 to D. C. Betjeman; 1984647 of Dec. 18, 1934 to A. G. Leonard, Jr.; 2069444 of Feb. 2, 1937 to H. H. Honigbaum; **Can. Pats.** 152981 of Jan. 6, 1914 to W. S. Orth; 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 201057 and 201058 of Jun. 15, 1920 to Flintkote Co.; 211668 and 211669 of May 17, 1921 to Roofing Patents Co.; 214178 of Nov. 8, 1921 to Roofing Patents Co.; 216538 of Mar. 7, 1922 to Roofing Patents Co.; 223888 of Sep. 19, 1922 to Roofing Patents Co.; 239433 of Apr. 11, 1924 to Guyton & Cumfer Mfg. Co.; 247334 of Mar. 3, 1925 to Flintkote Co.; 253523 of Sep. 8, 1925 to Guyton & Cumfer Mfg. Co.; 254365 of Oct. 6, 1925 to P. P. Welty; 259399 of Mar. 30, 1926 to Flintkote Co.; 263204 of Aug. 3, 1926 to Flintkote Co.; 266926 of Dec. 21, 1926 to Flintkote Co.; 268578 of Feb. 22, 1927 to Flintkote Co.; 294016 of Oct. 15, 1929 to Flintkote Co.; 303385 of Aug. 26, 1930 to Barrett Co.; 312825 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co ; 314865 of Sep. 1, 1931 to Patent & Licensing Corp.; 330051 of Feb. 7, 1933 to Patent & Licensing Corp.; 358757 of Jun. 30, 1936 to J. A. Topping; **Brit. Pats.** 138874, 138883 and 138892 of Dec. 28, 1917 to Flintkote Co.; 162424 of May 5, 1921 to Flintkote Co.; **Ger. Pats.** 24619 of Mar. 13, 1883 to Hermann Schulz; 226401 of Dec. 25, 1907 to Flintkote Mfg. Co.; **French Pat.** 517127 of Jun. 14, 1920 to Flintkote Co.

498 [p. 670] **U. S. Pats.** 1265105 of May 7, 1918 to F. C. Overbury; Designs 53086 and 53087 of Mar. 11, 1919 to F. C. Overbury.

499 [p. 670] **U. S. Pats.** 1277861 of Sep. 3, 1918 to C. M. Clarke; 1298535 and 1298536 of Mar. 25, 1919 to E. A. Mastick, Jr.; 1559109 of Oct. 27, 1925 to Lester Kirschbraun; 1596449 of Aug. 17, 1926 to C. E. Rahr; **Can. Pats.** 205281 and 205282 of Nov. 2, 1920 to E. A. Mastick, Jr.

500 [p. 670] **U. S. Pats.** 1745631 of Feb. 4, 1930 to A. O. Mickelson; 1777076 of Sep. 30, 1930 to H. A. Cumfer; **Brit. Pat.** 352815 of Sep. 20, 1930 to H. A. Cumfer and W. J. Mason.

501 [p. 670] **U. S. Pats.** 1207523 of Dec. 5, 1916 to S. M. Ford; 1213472 of Jan. 23, 1917 to S. M. Ford; 1356500 of Oct. 19, 1920 to Henry Wulff; 1381388 of Jun. 14, 1921 to Henry Wulff; 1424459 of Aug. 1, 1922 to S. M. Ford; Reissues 15565 and 15566 of Mar. 27, 1923 to Henry Wulff; 1457662 and 1457663 of Jun. 5, 1923 to S. M. Ford; 1463314 of Jul. 31, 1923 to S. M. Ford; 1498555 of Jun. 24, 1924 to H. J. Langan; 1517582 of Dec. 2, 1924 to C. E. Rahr; 1646963 of Oct. 25, 1927 to H. H. Honigbaum; 1840984 of Jan. 12, 1932 to C. G. Talbott; 1911141 of May 23, 1933 to A. C. Fischer; **Can. Pats.** 179757 and 179758 of Oct.

16, 1917 to S. M. Ford; 204021 of Sep. 14, 1920 to S. M. Ford; 208684 of Feb. 22, 1921 to S. M. Ford; 248998 of Apr. 28, 1925 to H. J. Langan; 269633 of Apr. 5, 1927 to Flintkote Co.; 279907 of May 8, 1928 to H. H. Honigbaum.

502 [p. 670] **U. S. Pats.** 1704058 of Mar. 5, 1929 to F. C. Overbury; 1759995 of May 27, 1930 to F. C. Overbury; **Can. Pat.** 261769 of Jun. 15, 1906 to Flintkote Co.

503 [p. 670] **U. S. Pat.** 1843370 of Feb. 2, 1932 to F. C. Overbury.

504 [p. 670] **U. S. Pats.** 1318238 of Oct. 7, 1919 to A. S. Speer; 1379727 of May 31, 1921 to A. S. Speer; Reissue 15496 of Nov. 21, 1922 to A. S. Speer; Reissues 16390 and 16391 of Jul. 20, 1926 to A. S. Speer; **Can. Pat.** 217363 of Mar. 28, 1922 to Roofing Patents Co.

505 [p. 671] **U. S. Pats.** 1285147 of Nov. 19, 1918 to W. A. Harris; 1447567 of Mar. 6, 1923 to F. C. Overbury; 1513365 of Oct. 28, 1924 to G. C. Blohm; 1724269 of Aug. 13, 1929 to Dozier Finley; 1741566 of Dec. 31, 1929 to W. A. Harris.

506 [p. 671] **U. S. Pats.** 1531151 of Mar. 24, 1925 to H. H. Steele; 1563131 of Nov. 24, 1925 to J. C. Whitmore; 1802868 of Apr. 28, 1931 to E. R. Black; 1894614 and 1894615 of Jan. 17, 1933 to J. L. Wettlaufer; 2001399 of May 14, 1935 to J. A. Scharwath; **Can. Pat.** 315859 of Oct. 6, 1931 to Black Systems, Inc.

507 [p. 671] **U. S. Pats.** 1325546 of Dec. 23, 1919 to H. R. Wardell; 1776949 of Sep. 30, 1930 to P. E. Lumbard.

508 [p. 671] **U. S. Pats.** 1345099 of Jun. 29, 1920 to F. C. Overbury; 1841296 of Jan. 12, 1932 to F. C. Overbury; **Can. Pat.** 330051 of Feb. 7, 1933 to Patent & Licensing Corp.

509 [p. 671] **U. S. Pats.** 1433983 of Oct. 31, 1922 to Frank Christenson; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1729212 of Sep. 24, 1929 to A. C. Fischer; 1824471 and 1824472 of Sep. 22, 1931 to A. C. Fischer; 1879378 of Sep. 27, 1932 to W. C. McWilliams; 1898989 and 1898990 of Feb. 21, 1933 to N. P. Harshberger; 1928285 of Sep. 26, 1933 to A. C. Fischer; 1936327 of Nov. 21, 1933 to A. C. Fischer.

510 [p. 671] **U. S. Pats.** 1433983 of Oct. 31, 1922 to Frank Christenson; 1516238 of Nov. 18, 1924 to C. W. Mortimer; 1536027 of Apr. 28, 1925 to C. W. Mortimer; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1729212 of Sep. 24, 1929 to A. C. Fischer; **Brit. Pat.** 113514 of Mar. 9, 1917 to H. J. Palmer.

511 [p. 671] **U. S. Pats.** 891501 of Jun. 23, 1908 to F. C. Overbury; 1348498 of Aug. 3, 1920 to C. L. Keller; 1348503 of Aug. 3, 1920 to A. R. Lukens, Jr.; 1464492 of Aug. 14, 1923 to Leon Busha; 1487662 of Mar. 18, 1924 to S. M. Langston; 1508365 of Sep. 9, 1924 to A. R. Lukens, Jr. and C. L. Keller; 1602007 of Oct. 5, 1926 to A. C. Fischer; 1614446 of Jan. 11, 1927 to A. R. Lukens, Jr.; 1629146 of May 17, 1927 to Leon Busha; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1702609 of Feb. 19, 1929 to E. W. Lesher; 1759901 of May 27, 1930 to N. P. Harshberger; 1862852 of Jun. 14, 1932 to N. P. Harshberger; 1885346 of Nov. 1, 1932 to N. P. Harshberger; Design 90115 of Jun. 13, 1933 to N. P. Harshberger; Designs 92379 and 92380 of May 29, 1934 to J. A. Topping; Design 93824 of Nov. 13, 1934 to N. P. Harshberger; Design 96547 of Aug. 13, 1935 to N. P. Harshberger; 2013391 of Sep. 3, 1935 to M. W. Searls; 2068118 of Jan. 19, 1937 to J. A. Topping; **Can. Pat.** 352106 of Aug. 6, 1935 to N. P. Harshberger; 354855 of Dec. 17, 1935 to J. A. Topping; 359910 of Aug. 18, 1936 to Patent & Licensing Corp.; 360875 of Sep. 29, 1936 to Edward Karfiol; 363128 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.

512 [p. 671] **U. S. Pats.** 1486346 of Mar. 11, 1924 to Cicero Hoskins; 1574098 of Feb. 23, 1926 to B. C. Kridler.

513 [p. 671] **U. S. Pat.** 1591042 of Jul. 6, 1926 to J. W. Ingels.

514 [p. 671] **U. S. Pats.** 1496108 of Jun. 3, 1924 to T. H. Wilson and E. R. Maltby; 1500709 of Jul. 8, 1924 to C. L. Keller.

515 [p. 671] **U. S. Pat.** 1570152 of Jan. 19, 1926 to J. E. Hooker.

516 [p. 671] **U. S. Pats.** 1479035 of Jan. 1, 1924 to A. C. Fischer; 1488447 of Mar. 25, 1924 to J. A. Topping; 1494788 of May 20, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1580096 of Apr. 6, 1926 to F. J. Ballintine; 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1709776 of Apr. 16, 1929 to Abbott Coburn; 1820388 of Aug. 25, 1931 to H. R. French; 1873944 of Aug. 23, 1932 to J. E. Black; 1898990 of Feb. 21, 1933 to N. P. Harshberger; 2060618 of Nov. 10, 1936 to H. H. Honigbaum.

517 [p. 671] **U. S. Pats.** 1524125 of Jan. 27, 1925 to L. M. Ford; 1544956 of Jul. 7, 1925 to C. T. Torbert; 1607357 of Nov. 16, 1926 to L. L. Martin and C. R. Mayfield; 1770693 of Jul. 15, 1930 to N. P. Harshberger; 1919148 of Jul. 18, 1933 to H. L. Guy.

518 [p. 671] **U. S. Pats.** 1494789 of May 20, 1924 to Herbert Abraham; 1524125 of Jan. 27, 1925 to L. M. Ford; 1548017 of Aug. 4, 1925 to Herbert Abraham; 1818850 of Aug. 11, 1931 to N. P. Harshberger.

519 [p. 671] **U. S. Pats.** 1356930 of Oct. 26, 1920 to J. R. McCord, Jr.; 1446858 of Feb. 27, 1923 to Max Rachlin; 1480167 of Jan. 8, 1924 to B. C. Kridler and J. C. Boyle; 1483735 of Feb. 12, 1924 to B. C. Kridler and J. C. Boyle; 1493852 of May 13, 1924 to W. A. Fogg; 1494707 of May 20, 1924 to Max Rachlin; 1498947 of Jun. 24, 1924 to J. O. Bewan; 1510497 of Oct. 7, 1924 to C. L. Keller; 1510756 of Oct. 7, 1924 to L. T. Ayrault and John Ayrault, Jr.; 1538329 of May 19, 1925 to H. H. Honigbaum; 1570222 of Jan. 19, 1926 to J. C. Barley; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1623127 of Apr. 5, 1927 to A. E. F. Moone; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1744589 of Jan. 21, 1930 to H. N. Striewig; 1898989 of Feb. 21, 1933 to N. P. Harshberger; 1925939 of Sep. 5, 1933 to Irving Sherman; 2007855 of Jul. 9, 1935 to H. L. Guy; **Can. Pats.** 262603 of Jul. 13, 1926 to Beaver Co., Ltd.; 286850 of Jan. 29, 1929 to Lehon Co.; 310683 of Apr. 21, 1931 to Lehon Co.

520 [p. 671] **U. S. Pat.** 1570152 of Jan. 19, 1926 to J. E. Hooker.

521 [p. 671] **U. S. Pats.** 1209955 of Dec. 26, 1916 to S. M. Ford; 1415175 of May 9, 1922 to H. G. Hose; 1580096 of Apr. 6, 1926 to F. J. Ballintine; 1596449 of Aug. 17, 1926 to C. E. Rahr; 1597135 of Aug. 24, 1926 to Lester Wittenberg; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1705497 of Mar. 19, 1929 to F. C. Overbury; 1802868 of Apr. 28, 1931 to E. R. Black; 1927436 of Sep. 19, 1933 to A. C. Fischer; **Can. Pats.** 248536 of Apr. 7, 1925 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.

522 [p. 671] **U. S. Pat.** 1402361 of Jan. 3, 1922 to H. G. Hose.

523 [p. 672] **U. S. Pats.** 1198653 of Sep. 19, 1916 to F. C. Overbury; 1582266 of Apr. 27, 1926 to N. P. Harshberger; 1596272 of Aug. 17, 1926 to G. M. Jordan; 1959519 of May 22, 1934 to E. R. Black; 1975986 of Oct. 9, 1934 to S. C. Straub; **Can. Pat.** 244166 of Nov. 4, 1924 to Charles Southgate.

524 [p. 672] **U. S. Pats.** 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1681975 of Aug. 28, 1928 to Isadore Davis.

525 [p. 672] **U. S. Pats.** 1063710 of Jun. 3, 1913 to W. F. McKay; 1219652 of Mar. 20, 1917 to W. F. McKay; 1426497 of Aug. 22, 1922 to O. K. Outman; 1451369 of Apr. 10, 1923 to R. P. Perry; 1455713 of May 15, 1923 to D. A. Cumfer; 1478544 of Dec. 25, 1923 to R. W. Bird; 1501512 of Jul. 15, 1924 to O. A. Bigler; 1534165 of Apr. 21, 1925 to D. A. Cumfer; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1593594 of Jul. 27, 1926 to R. P. Perry; 1627665 of May 10, 1927 to F. C. Overbury; 1729212 of Sep. 24, 1929 to A. C. Fischer; 1969074 of Aug. 7, 1934 to N. P. Harshberger; **Can. Pats.** 175819 of Mar. 20, 1917 to W. F. McKay; 248537 of Apr. 7, 1925 to Flintkote Co.; 249587 of May 12, 1925 to Flintkote Co.; 251519 of Jul. 7, 1925 to Flintkote Co.; 256458 of Dec. 22, 1925 to H. A. Cumfer.

526 [p. 672] **U. S. Pats.** 1114032 of Oct. 20, 1914 to J. R. Powell and B. G. Casler; 1415923 of May16, 1922 to R. B. Crabbs; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1659808 of Feb. 21, 1928 to M. L. Caton; 1697464 of Jan. 1, 1929 to W. T. Hofmann; 1773117 of Aug. 19, 1930 to C. E. Rahr; **Can. Pats.** 229986 of Apr. 3, 1923 to W. T. Hofmann; 244197 of Nov. 4, 1924 to Barrett Co.; 249353 of May 5, 1925 to Flintkote Co.; 329238 of Jan. 10, 1933 to Certainteed Products Corp.; 332651 of May 23, 1933 to Certainteed Products Corp.; 335778 of Sep. 19, 1933 to Certainteed Products Corp.

527 [p. 672] **U. S. Pats.** 1154334 of Sep. 21, 1915 to F. C. Overbury; 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; 1264831 of Apr. 30, 1918 to W. F. McKay; 1295360 of Feb. 25, 1919 to F. C. Overbury; 1296984 of Mar. 11, 1919 to O. D. McFarland; 1345627 of Jul. 6, 1920 to F. C. Overbury; Reissue 15489 of Nov. 14, 1922 to O. D. McFarland; 1456224 of May 22, 1923 to A. E. Currier; 1650285 of Nov. 22, 1927 to L. F. Lindley; 1898989 and 1898990 of Feb. 21, 1933 to N. P. Harshberger; **Can. Pat.** 223401 of Sep. 5, 1922 to Roofing Patents Co.; **Brit. Pat.** 138875 of Jan. 21, 1915 to Flintkote Co.

528 [p. 672] **U. S. Pats.** 1623189 of Apr. 5, 1927 to Lester Kirschbraun; 1673991 of Jun.

19, 1928 to F. C. Overbury; 1756742 of Apr. 29, 1930, to N. P. Harshberger and R. P. Harshberger; 1958568 of May 15, 1934 to H. E. Beckman; **Can. Pat.** 260003 of Apr. 20, 1926 to F. C. Overbury.

529 [p. 672] **U. S. Pats.** 1365902 of Jan. 18, 1921 to S. M. Ford; 1619600 of Mar. 1, 1927 to H. A. Cumfer; 1795913 of Mar. 10, 1931 to W. W. Weaver; 2064473 of Dec. 15, 1936 to R. A. Holdsworth.

530 [p. 672] **U. S. Pat.** 1613101 of Jan. 4, 1927 to N. P. Harshberger.

531 [p. 672] **U. S. Pats.** 1398250 of Nov. 29, 1921 to E. J. Yetter; 1469555 of Oct. 2, 1923 to H. A. Cumfer; 1475595 of Nov. 27, 1923 to C. E. Rahr and R. T. Pollock; 1484761 of Feb. 26, 1924 to H. A. Cumfer; 1512248 of Oct. 21, 1924 to James Smith and M. E. Constable; 1513940 of Nov. 4, 1924 to James Smith and M. E. Constable; 1517860 of Dec. 2, 1924 to C. E. Rahr and R. T. Pollock; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1595243 of Aug. 10, 1926 to R. T. Pollock; 1602007 of Oct. 5, 1926 to A. C. Fischer; 1699213 of Jan. 15, 1929 to C. A. Statler; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1779175 of Oct. 21, 1930 to L. F. Lindley; 1913836 of Jun. 13, 1933 to M. L. Hamlin; 1993086 of Mar. 5, 1935 to S. W. Chaffee; **Can. Pats.** 249587 of May 12, 1925 to Flintkote Co.; 251355 of Jul. 7, 1925 to H. A. Cumfer; 256458 of Dec. 22, 1925 to H. A. Cumfer; 263005 of Jul. 27, 1926 to Flintkote Co.

532 [p. 672] **U. S. Pat.** 1759901 of May 27, 1930 to N. P. Harshberger.

533 [p. 672] **U. S. Pat.** 1126114 of Jan. 26, 1914 to A. S. Spiegel.

534 [p. 673] See Reference 447.

535 [p. 673] **U. S. Pats.** 1495070 of May 20, 1924 to Dozier Finley; 1840997 of Jan. 12, 1932 to F. W. Yeager; 1843370 of Feb. 2, 1932 to F. C. Overbury; 1860899 of May 31, 1932 to T. D. Miller; 1870414 of Aug. 9, 1932 to H. L. Levin and F. H. Neher; 1937933 of Dec. 5, 1933 to F. W. Yeager; 1974047 of Sep. 18, 1934 to N. P. Harshberger; **Can. Pats.** 242873 of Sep. 9, 1924 to Dozier Finley; 314865 of Sep. 1, 1931 to Patent & Licensing Corp.; 320408 of Mar. 8, 1932 to Patent & Licensing Corp.; 323534 of Jun. 21, 1932 to Patent & Licensing Corp.; 337158 of Nov. 14, 1933 to Patent & Licensing Corp.; 346638 of Dec. 11, 1934 to Barrett Co.

536 [p. 673] **U. S. Pats.** 1126114 of Jan. 26, 1914 to A. S. Spiegel; 1221370 of Apr. 3, 1917 to F. C. Overbury and H. C. Platts; 1265226 of May 7, 1918 to E. A. Mastick; 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1469543 of Oct. 2, 1923 to G. I. Strachan and J. L. Strachan; **Can. Pat.** 210451 of Apr. 12, 1921 to Roofing Patents Co.

537 [p. 673] **U. S. Pat.** 1994643 of Mar. 19, 1935 to N. P. Harshberger; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2024861 of Dec. 17, 1935 to H. H. Honigbaum; **Can. Pat.** 364943 of Mar. 23, 1930 to Certainteed Products Corp.

538 [p. 673] **U. S. Pats.** 194766 of Sep. 4, 1877 to Chester Comstock (metal); 520370 of May 22, 1894 to W. H. Mullins (metal); 1243064 of Oct. 16, 1917 to O. A. Heppes; 1290555 of Jan. 7, 1919 to O. A. Heppes; 1317384 of Sep. 30, 1919 to F. C. Overbury; Design. 54917 of Apr. 20, 1920 to O. A. Heppes; 1447567 of Mar. 6, 1923 to F. C. Overbury; 1473919 of Nov. 13, 1923 to Herbert Abraham; 1973863 of Sep. 18, 1934 to D. C. Betjeman; **Can. Pats.** 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 170312 of Jun. 20, 1916 to O. A. Heppes; 347985 of Feb. 5, 1935 to Bakelite Building Products Co.; **Swiss Pat.** 5035 of May 9, 1892 to Johann Eberli (wood).

539 [p. 673] **U. S. Pat.** 1295360 of Feb. 25, 1919 to F. C. Overbury; **Can. Pat.** 347571 of Jan. 15, 1935 to Self-Locking Carton Co.

540 [p. 673] **U. S. Pat.** 1345627 of Jul. 6, 1920 to F. C. Overbury.

541 [p. 673] **U. S. Pats.** 1729212 of Sep. 24, 1929 to A. C. Fischer; 1824471 of Sep. 22, 1931 to A. C. Fischer; 1936327 of Nov. 21, 1933 to A. C. Fischer.

542 [p. 673] **U. S. Pats.** 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1851088 of Mar. 29, 1932 to J. H. Duncan; 1919148 of Jul. 18, 1933 to H. L. Guy.

543 [p. 673] **U. S. Pats.** 978334 of Dec. 13, 1911 to F. C. Overbury; **Ger. Pat.** 252191 of Nov. 16, 1910 to Flintkote Mfg. Co.

544 [p. 675] **U. S. Pats.** 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1108236 of Aug. 25, 1914 to H. M. Reynolds; 1126932 of Feb. 2, 1915 to Herbert Abraham; 1445161 of Feb.

13, 1923 to Jacob Ott; 1500709 of Jul. 8, 1924 to C. L. Keller; Design 68522 of Oct. 20, 1925 to C. W. Dohm; 1596449 of Aug. 17, 1926 to C. E. Rahr; 1604339 of Oct. 26, 1926 to H. A. Cumfer; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1682921 of Sep. 4, 1928 to O. D. McFarland; 1687873 of Oct. 16, 1928 to O. D. McFarland; 1758410 of May 13, 1930 to Hugo Reichel; 1766244 of Jun. 24, 1930 to H. A. Cumfer; 1773352 of Aug. 19, 1930 to Dozier Finley; 1846635 of Feb. 23, 1932 to Dozier Finley; 1851088 of Mar. 29, 1932 to J. H. Duncan; 1973863 of Sep. 18, 1934 to D. C. Betjeman; 2006417 of Jul. 2, 1935 to J. A. Topping; **Can. Pats.** 139981 of Apr. 23, 1912 to J. C. Searle (metal); 237758 of Feb. 12, 1924 to Jacob Ott; 248539 of Apr. 7, 1925 to Flintkote Co.; 249094 of Apr. 28, 1925 to Flintkote Co.; 263204 of Aug. 3, 1926 to Flintkote Co.; 300349 of May 20, 1930 to Barrett Co.; 312824 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co.; 334298 of Jul. 25, 1933 to Barrett Co.; 352256 of Aug. 6, 1935 to J. A. Topping; **Ger. Pat.** 249986 of Aug. 30, 1910 to Naumann Schefftel; **Austrian Pats.** 48947 of Jul. 10, 1911 to Heinrich Schwarz; 53358 of May 12, 1912 to Heinrich Schwarz;

545 [p. 675] **U. S. Pats.** 1318238 of Oct. 7, 1919 to A. S. Speer; 1352154 of Sep. 7, 1920 to A. S. Speer; 1379727 of May 31, 1921 to A. S. Speer; Reissue 15496 of Nov. 21, 1922 to A. S. Speer; 1473919 of Nov. 13, 1923 to Herbert Abraham; 1486346 of Mar. 11, 1924 to Cicero Hoskins; Reissue 15955 of Nov. 25, 1924 to A. S. Speer; Reissue 16390 of Jul. 20, 1926 to A. S. Speer; 1831374 of Nov. 10, 1931 to J. A. Topping; **Can. Pats.** 201253 of Apr. 12, 1921 to Herbert Abraham; 211666 of May 17, 1921 to Roofing Patents Co.; 217363 of Mar. 28, 1922 to Roofing Patents Co.; **Brit. Pat.** 169874 of Oct. 13, 1921 to Herbert Abraham.

546 [p. 675] **U. S. Pats.** 1546868 of Jul. 21, 1925 to H. A. Sjodahl; Design 87104 of Jun. 7, 1932 to C. R. Eckert; 1956381 of Apr. 24, 1934 to C. R. Eckert; 1974047 of Sep. 18, 1934 to N. P. Harshberger; Design 104095 of Apr. 13, 1937 to J. R. Fife; **Can. Pat.** 263205 of Aug. 3, 1926 to Flintkote Co.; 363128 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.

547 [p. 675] **U. S. Pats.** 1729212 of Sep. 24, 1929 to A. C. Fischer; 1802378 of Apr. 28, 1931 to N. P. Harshberger; 1824471 of Sep. 22, 1931 to A. C. Fischer; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1936327 of Nov. 21, 1933 to A. C. Fischer; 1956285 of Apr. 24, 1934 to N. P. Harshberger; Reissue 19903 of Mar. 24, 1936 to N. P. Harshberger.

548 [p. 675] **U. S. Pats.** 1655885 of Jan. 10, 1928 to Reid Adair; 1732403 of Oct. 22, 1929 to W. A. Harris and H. R. French; Design 81768 of Aug. 5, 1930 to J. A. Topping; Design 88995 of Jan. 10, 1933 to R. J. Tobin; **Can. Pats.** 278369 and 278370 of Mar. 6, 1928 to Flintkote Co.; 284974 of Nov. 20, 1928 to Flintkote Co.

549 [p. 675] **U. S. Pats.** 341188 of May 4, 1886 to Ezekiel Van Noorden (metal); 1453373 of May 1, 1923 to Herbert Abraham; 1509795 of Sep. 23, 1924 to J. F. Tiner (metal); 1524125 of Jan. 27, 1925 to L. M. Ford; 1587017 of Jun. 1, 1926 to Hugh MacInnes; 1613102 of Jan. 4, 1927 to N. P. Harshberger; 1656434 of Jan. 17, 1928 to August Gietz; 1670469 of May 22, 1928 to J. A. McCarthy; 1721242 of Jul. 16, 1929 to J. P. Wolff; 1729846 of Oct. 1, 1929 to F. E. Theilacker; 1756741 and 1756743 of Apr. 29, 1930 to N. P. Harshberger; 1770693 of Jul. 15, 1930 to N. P. Harshberger; Reissue 17836 of Oct. 21, 1930 to J. A. McCarthy; 1956732 of May 1, 1934 to W. P. Schulz; **Can. Pat.** 362432 of Dec. 8, 1936 to Barrett Co.; **Brit. Pat.** 203988 of Feb. 27, 1923 to Herbert Abraham.

550 [p. 675] **U. S. Pats.** 1356930 of Oct. 26, 1920 to J. R. McCord, Jr.; 1463632 of Jul. 31, 1923 to R. S. Severns; 1570222 of Jan. 19, 1926 to J. C. Barley; 1574099 of Feb. 23, 1926 to B. C. Kridler; 1612718 of Dec. 28, 1926 to J. F. Grice; 1623127 of Apr. 5, 1927 to A. E. F. Moone; 1627429 of May 3, 1927 to Isadore Davis; 1634465 of Jul. 5, 1927 to H. G. Kelly; Design 75433 of Jun. 5, 1928 to H. G. Kelly; 1744589 of Jan. 21, 1930 to H. N. Strieweg; 1768134 of Jun. 24, 1930 to A. E. F. Moone; 1775937 of Sep. 16, 1930 to C. L. Keller; 1814405 of Jul. 14, 1931 to Alvin Myhre; 1980053 of Nov. 6, 1934 to N. P. Harshberger; 2037163 of Apr. 14, 1936 to H. L. Guy; **Can. Pat.** 310683 of Apr. 21, 1931 to Lehon Co.

551 [p. 675] **U. S. Pats.** 1402361 of Jan. 3, 1922 to H. G. Hose; 1453373 of May 1, 1923 to Herbert Abraham; 1559506 of Oct. 27, 1925 to William Freegard; 1619601 of Mar. 1, 1927 to H. A. Cumfer; 1659575 of Feb. 21, 1928 to George Ritter; 1677492 of Jul. 17, 1928 to George Ritter; 1731187 of Oct. 8, 1929 to R. B. Adams; 1771990 of Aug. 5, 1930 to J. C. Bergner; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1993163, 1993164, 1993165 and 1993166 of Mar. 5, 1935 to N. P. Harshberger; Reissue 19903 of Mar. 24, 1936 to N. P.

Harshberger; **Can. Pats.** 248535 of Apr. 7, 1923 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.; 311390 of May 19, 1931 to R. B. Adams; **Brit. Pat.** 203988 of Feb. 27, 1923 to Herbert Abraham.

552 [p. 675] **U. S. Pats.** 1681975 of Aug. 28, 1928 to Isadore Davis; 1685493 of Sep. 25, 1928 to H. G. Kelly; 1741515 of Dec. 31, 1929 to Martin Halprin; 1885346 of Nov. 1, 1932 to N. P. Harshberger; 2017230 of Oct. 15, 1935 to E. R. Black.

553 [p. 675] **U. S. Pats.** 1868751 of Jul. 26, 1932 to H. C. Koch; 1898989 of Feb. 21, 1933 to N. P. Harshberger; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1945485 of Jan. 30, 1934 to N. P. Harshberger; Reissue 19903 of Mar. 24, 1936 to N. P. Harshberger; **Can. Pat.** 351714 of Jul. 16, 1935 to Patent & Licensing Corp.

554 [p. 675] **U. S. Pats.** 1602007 of Oct. 5, 1926 to A. C. Fischer; 1602164 of Oct. 5, 1926 to F. C. Overbury; 1619598 of Mar. 1, 1927 to H. A. Cumfer; 1619601 of Mar. 1, 1927 to H. A. Cumfer; 1627429 of May 3, 1927 to Isadore Davis; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1802378 of Apr. 28, 1931 to N. P. Harshberger; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2041761 of May 26, 1936 to N. P. Harshberger; **Can. Pats.** 248535 of Apr. 7, 1925 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.

555 [p. 675] **U. S. Pats.** 1949907 of Mar. 6, 1934 to N. P. Harshberger; Design 91654 of Mar. 6, 1934 to N. P. Harshberger.

556 [p. 675] **U. S. Pats.** 373373 of Nov. 15, 1867 to L. H. Montross (metal); 512986 of Jan. 16, 1894 to L. H. Montross (metal); 668625 of Feb. 26, 1901 to C. W. Connor (metal); 820294 of May 8, 1906 to Albert Friedley (metal); 1150298 of Aug. 17, 1915 to F. C. Overbury; 1257321 of Feb. 26, 1916 to H. A. Cumfer and O. D. McFarland; 1340402 of May 18, 1920 to F. C. Overbury; 1583977 of May 11, 1926 to H. G. Kelly; 1776949 of Sep. 30, 1930 to P. E. Lumbard; 1834004 of Dec. 1, 1931 to F. C. Overbury and O. A. Heppes; **Can. Pats.** 69189 of Oct. 30, 1900 to C. W. Connor (metal); 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 347571 of Jan. 15, 1935 to Self-Locking Carton Co.

557 [p. 675] **U. S. Pats.** 1326899 of Jan. 6, 1920 to Herbert Abraham; Reissue 15280 of Feb. 7, 1922 to Herbert Abraham; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1482776 of Feb. 5, 1924 to Herbert Abraham; 1548107 of Aug. 4, 1925 to E. T. Street; 1593594 of Jul. 27, 1926 to R. P. Perry; **Can. Pats.** 199466 of Apr. 27, 1920 to Herbert Abraham; 219560 of Jun. 13, 1922 to Ruberoid Co., Ltd.; 233887 of Aug. 28, 1923 to Ruberoid Co., Ltd.; 236139 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 242500 of Aug. 26, 1924 to Ruberoid Co., Ltd.; 285501 of Dec. 11, 1928 to Barrett Co.; 303385 of Aug. 26, 1930 to Barrett Co.; **Brit. Pat.** 152858 of Oct. 13, 1919 to Herbert Abraham.

558 [p. 675] **U. S. Pats.** 1412295 of Apr. 11, 1922 to A. S. Speer; 1455232 of May 15, 1923 to A. S. Speer; Reissue 16429 of Sep. 21, 1926 to A. S. Speer; 1623983 of Apr. 12, 1927 to A. S. Speer; **Can. Pat.** 217361 of Mar. 28, 1922 to Roofing Patents Co.

559 [p. 675] **U. S. Pats.** 1415758 of May 9, 1922 to Herbert Abraham; 1557391 of Oct. 13, 1925 to Herbert Abraham; **Can. Pats.** 219560 of Jun. 13, 1922 to Herbert Abraham; 233150 of Jul. 31, 1923 to Ruberoid Co., Ltd.; 242295 of Aug. 19, 1924 to Ruberoid Co., Ltd.

560 [p. 675] **U. S. Pats.** 1729212 of Sep. 24, 1929 to A. C. Fischer; 1824471 of Sep. 22, 1931 to A. C. Fischer; 1936327 of Nov. 21, 1933 to A. C. Fischer.

561 [p. 675] **U. S. Pats.** Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; 1464493 and 1464494 of Aug. 14, 1923 to Leon Busha; 1500709 of Jul. 8, 1924 to C. L. Keller; 1533969 of Apr. 14, 1925 to Leon Busha; Reissue 16621 of May 10, 1927 to Leon Busha; 1927436 of Sep. 19, 1933 to A. C. Fischer; **Can. Pat.** 259057 of Mar. 16, 1926 to W. E. Nelson.

562 [p. 675] **U. S. Pats.** 1442614 of Jan. 16, 1923 to J. E. Hooker; 1510533 of Oct. 7, 1924 to Herbert Abraham; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1756741 of Apr. 29, 1930 to N. P. Harshberger and R. P. Harshberger.

563 [p. 675] **U. S. Pats.** 1494707 of May 20, 1924 to Max Rachlin; 1544392 of Jun. 30, 1925 to V. J. Harward and W. P. Budd; Design 75433 of Jun. 5, 1928 to H. G. Kelly.

564 [p. 675] **U. S. Pats.** 1659575 of Feb. 21, 1928 to George Ritter; 1795277 of Mar. 3, 1931 to A. C. Fischer.

565 [p. 675] **U. S. Pat.** 1885346 of Nov. 1, 1932 to N. P. Harshberger; **Can. Pat.** 244166 of Nov. 4, 1924 to Charles Southgate.

566 [p. 678] **U. S. Pats.** 1466077 of Aug. 28, 1923 to H. R. Wardell; 1584343 of May 11, 1926 to Herbert Abraham; **Can. Pats.** 236136 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 261144 of May 25, 1926 to Johns-Manville, Inc.

567 [p. 678] **U. S. Pats.** 1619599 of Mar. 1, 1927 to H. A. Cumfer; 2024861 of Dec. 17, 1935 to H. H. Honigbaum.

568 [p. 678] **U. S. Pats.** 346209 of Jul. 27, 1886 to Frederick Mankey (wood); 512986 of Jan. 16, 1894 to L. H. Montross (wood;); 520370 of May 22, 1894 to W. H. Mullins (metal); Design 24082 of Mar. 5, 1895 to W. H. Mullins (metal); 1295360 of Feb. 25, 1919 to F. C. Overbury; Design 54160 of Nov. 4, 1919 to R. L. Woodruff; 1455232 of May 15, 1923 to A. S. Speer; 1473919 of Nov. 13, 1923 to Herbert Abraham; Reissue 16429 of Sep. 21, 1926 to A. S. Speer; Design 74669 of Mar. 13, 1928 to J. R Fife; 1729212 of Sep. 24, 1929 to A. C. Fischer; 1974047 of Sep. 18, 1934 to N. P. Harshberger; Designs 99248 and 99249 of Apr. 7, 1936 to J. J. Piazza; **Can. Pats.** 46251 of Jun. 5, 1894 to H. D. Walker (metal); 67812 of Jun. 21, 1900 to H. D. Walker (metal); 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 341469 of May 8, 1934 to Building Products, Ltd.

569 [p. 678] **U. S. Pats.** 891501 of Jun. 23, 1908 to F. C. Overbury; 1274623 of Aug. 6, 1918 to A. S. Spiegel; 1301964 of Apr. 29, 1919 to F. C. Overbury; 1345627 of Jul. 6; 1920 to F. C. Overbury; 1447567 of Mar. 6, 1923 to F. C. Overbury; 1473919 of Nov. 13, 1923 to Herbert Abraham; Design 67565 of Jun. 16, 1925 to A. W. Abbey and C. W. Dohm; 1599253 of Sep. 7, 1926 to Max Skolnik; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1670942 of May 22, 1928 to Walter Thomsen; Design 75612 of Jun. 26, 1928 to L. C. Cotner; Design 81712 of Jul. 29, 1930 to A. E. F. Moone; 1773352 of Aug. 19, 1930 to Dozier Finley; Design 84668 of Jul. 14, 1931 to S. H. Ralph; Designs 85638 and 85639 of Dec. 1, 1931 to Dozier Finley; Design 86085 of Jan. 26, 1932 to H. L. Guy; 1846635 of Feb. 23, 1932 to Dozier Finley; Design 87057 of May 31, 1932 to H. L. Guy; 1885346 of Nov. 1, 1932 to N. P. Harshberger; Design 89639 of Apr. 18, 1933 to D. B. Humphrey; Design 89783 of May 2, 1933 to A. E. F. Moone; 1913786 of Jun. 13, 1933 to A. E. F. Moone; Design 92504 of Jun. 12, 1934 to J. A. Topping; Design 92632 of Jun. 26, 1934 to J. A. Topping; Design 93191 of Aug. 28, 1934 to J. A. Topping; 2006417 of Jul. 2, 1935 to J. A. Topping; 2018718 of Oct. 29, 1935 to N. P. Harshberger; **Can. Pats.** 205148 of Oct. 26, 1920 to Flintkote Co.; 220782 of Jul. 11, 1922 to Roofing Patents Co.; 352770 of Sep. 3, 1935 to Certainteed Products Corp.; 364727 of Mar. 16, 1937 to J. A. Topping; 365088 of Mar. 30, 1937 to Bakelite Building Products Co., Inc.; **Austrian Pat.** 48947 of Jul. 10, 1911 to Heinrich Schwarz; **Swiss Pat.** 5035 of May 9, 1892 to Johann Eberli (wood).

570 [p. 678] **U. S. Pats.** 1641858 of Sep. 8, 1927 to J. A. McCarthy; 1705497 of Mar. 19, 1929 to F. C. Overbury; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1919148 of Jul. 18, 1933 to H. L. Guy.

571 [p. 678] **U. S. Pats.** 742614 of Oct. 27, 1903 to J. L. M. Du Four; 1683016 of Sep. 4, 1928 to E. L. Bell and O. M. Beebe.

572 [p. 678] **U. S. Pat.** 1345627 of Jul. 6, 1920 to F. C. Overbury.

573 [p. 678] **U. S. Pats.** 1633474 of Jun. 21, 1927 to Leon Busha; 1688917 of Oct. 23, 1928 to Leon Busha; Design 76843 of Nov. 6, 1928 to J. A. Topping; 1812654 of Jun. 30, 1931 to Lester Kirschbraun; 1913836 of Jun. 13, 1933 to M. L. Hamlin; 1975487 of Oct. 2, 1934 to J. A. Topping; **Can. Pats.** 330050 of Feb. 7, 1933 to Patent & Licensing Corp.; 355399 of Jan. 14, 1936 to J. A. Topping.

574 [p. 678] **U. S. Pats.** Design 61363 of Aug. 15, 1922 to N. Z. Butterick; 1445991 of Feb. 20, 1923 to N. Z. Butterick; 1701640 of Feb. 12, 1929 to F. E. Sherriff; 1741539 of Dec. 31, 1929 to J. M. Richardson; 1812654 of Jun. 30, 1931 to Lester Kirschbraun; 1863178 of Jun. 14, 1932 to H. R. Wood; **Can. Pats.** 232178 of Jun. 26, 1923 to N. Z. Butterick; 313515 of Jul. 21, 1931 to Patent & Licensing Corp.; **Brit. Pat.** 195097 of Mar. 20, 1922 to N. Z. Butterick.

575 [p. 678] **U. S. Pat.** 1812424 of Jun. 30, 1931 to H. R. Wood.

576 [p. 678] **U. S. Pat.** Design 83704 of Mar. 17, 1931 to G. R. Stark.

577 [p. 678] **U. S. Pats.** Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; 1442614 of Jan. 16, 1923 to J. E. Hooker; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker.

578 [p. 678] **U. S. Pats.** Design 74467 of Feb. 14, 1928 to Herbert Abraham; Design 77213 of Dec. 18, 1928 to Herbert Abraham.

579 [p. 678] **U. S. Pats.** 1555441 of Sep. 29, 1925 to H. A. Sjodahl; Design 68552 of Oct. 20, 1925 to H. A. Sjodahl; Designs 70988 and 70989 of Aug. 31, 1926 to H. A. Sjodahl; Designs 75761 and 75762 of Jul. 17, 1928 to Herbert Abraham; 1768280 of Jun. 24, 1930 to Salvatore Arcidiacono; 1843370 of Feb. 2, 1932 to F. C. Overbury; **Can. Pats.** 266300 of Nov. 30, 1926 to Chatfield Mfg. Co.; Designs 7620 and 7621 of Jun. 28, 1927 to H. A. Sjodahl.

580 [p. 678] **U. S. Pat.** Design 83718 of Mar. 24, 1931 to H. D. Brown.

581 [p. 678] **U. S. Pat.** 1843370 of Feb. 2, 1932 to F. C. Overbury.

582 [p. 678] **U. S. Pats.** 1442614 of Jan. 16, 1923 to J. E. Hooker; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker; Designs 70986 and 70987 of Aug. 31, 1926 to H. A. Sjodahl; 1927436 of Sep. 19, 1933 to A. C. Fischer; 1935656 of Nov. 21, 1933 to C. W. Mortimer; 1936327 of Nov. 21, 1933 to A. C. Fischer; Design 101732 of Oct. 27, 1936 to O. B. Clow; **Can. Pats.** 267307 of Jan. 4, 1927 to Chatfield Mfg. Co.; Designs 7618 and 7619 of Jun. 28, 1927 to H. A. Sjodahl.

583 [p. 678] **U. S. Pats.** Design 86194 of Feb. 16, 1932 to H. D. Brown; Design 91061 of Nov. 21, 1933 to J. R. Fife; Design 92132 of May 1, 1934 to R. A. Holdsworth.

584 [p. 678] **U. S. Pat.** Design 89471 of Mar. 14, 1933 to R. S. Streeter and O. E. Hendrick.

585 [p. 678] **U. S. Pats.** Design 57126 of Feb. 22, 1921 to O. A. Heppes; 1475551 of Nov. 27, 1923 to F. C. Overbury; 1745631 of Feb. 4, 1930 to A. O. Mickelson; **Can. Pats.** 244166 of Nov. 4, 1924 to Charles Southgate; 256566 of Dec. 22, 1925 to Flintkote Co.

586 [p. 678] **U. S. Pat.** 1489921 of Apr. 8, 1924 to T. J. Brown; 2037163 of Apr. 14, 1936 to H. L. Guy; **Can. Pats.** 256795 and 256796 of Dec. 29, 1925 to Flintkote Co.

587 [p. 678] **U. S. Pat.** 1257321 of Feb. 26, 1918 to H. A. Cumfer and O. D. McFarland.

588 [p. 678] **U. S. Pat.** 1745631 of Feb. 4, 1930 to A. O. Mickelson.

589 [p. 678] **Can. Pat.** 273858 of Sep. 13, 1927 to Flintkote Co.

590 [p. 678] **U. S. Pats.** 1326899 of Jan. 6, 1920, to Herbert Abraham; Reissue 15280 of Feb. 7, 1922 to Herbert Abraham; **Can. Pat.** 244166 of Nov. 4, 1924 to Charles Southgate.

591 [p. 681] **U. S. Pats.** 1157665 of Oct. 26, 1915 to M. B. Becker; 1174960 of Mar. 14, 1916 to M. B. Becker; 1181827 of May 2, 1916 to C. S. Bird; 1256508 of Feb. 19, 1918 to M. B. Becker; 1263987 of Apr. 23, 1918 to G. R. Wyman; 1270654 of Jan. 25, 1918 to F. C. Overbury; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1295361 of Feb. 25, 1919 to F. C. Overbury; 1314476 and 1314477 of Aug. 26, 1919 to F. C. Overbury; 1368947 of Feb. 15, 1921 to W. W. Levis; 1501512 of Jul. 15, 1924 to O. A. Bigler; 1515530 of Nov. 11, 1924 to P. P. Welty; 1546782 of Jul. 21, 1925 to L. M. Ford; 1563245 of Nov. 24, 1925 to P. P. Welty; 1599512 of Sep. 14, 1926 to W. H. Cady; 1612776 of Dec. 28, 1928 to Lester Kirschbraun; 1722702 of Jul. 30, 1929 to Lester Kirschbraun; 1755049 of Apr. 15, 1930 to W. H. Cady; 1756989 of May 6, 1930 to F. C. Overbury; 1760873 of Jun. 3, 1930 to Lester Kirschbraun; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1781877 of Nov. 18, 1930 to H. L. Levin; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1802032 of Apr. 21, 1931 to F. C. Overbury; 1807918 of Jun. 2, 1931 to Lester Kirschbraun and O. A. Heppes; Design 84808 of Aug. 11, 1931 to L. E. Calkins; 1826674 of Oct. 6, 1931 to B. H. Roberts and Erich Gach; 1843056 of Jan. 26, 1932 to P. P. Welty; 1860180 and 1860181 of May 24, 1932 to F. E. Horne; 1862627 of Jun. 14, 1932 to Robert Maclean; 1864806 of Jun. 28, 1932 to D. A. Cumfer; 1865579 of Jul. 5, 1932 to C. R. MacDonald; 1870426 of Aug. 9, 1932 to G. J. Snyder; 1872185 and 1872186 of Aug. 16, 1932 to R. W. B. Reade; 1873209 of Aug. 23, 1932 to C. R. MacDonald; 1873213 of Aug. 23, 1932 to F. C. Overbury; 1890017 of Dec. 6, 1932 to C. R. MacDonald; 1890018 of Dec. 6, 1932 to F. C. Overbury; 1905553 of Apr. 25, 1933 to R. D. Falk; 1908313 of May 9, 1933 to H. D. Brown; 1911014 of May 23, 1933 to C. P. Cowan; 1931554 of Oct. 24, 1933 to A. E. F. Moone;

1942449 of Jan. 9, 1934 to F. H. Rahr; 1943686 of Jan. 16, 1934 to O. V. McGrew; 1958571 and 1958572 of May 15, 1934 to F. H. Gilchrist; 1959519 of May 22, 1934 to E. R. Black; 1968535 of Jul. 31, 1934 to A. E. F. Moone; 1972810 of Sep. 4, 1934 to J. L. Wettlaufer; 1975324 of Oct. 2, 1934 to G. P. Jordan; 1976662 of Oct. 9, 1934 to J. A. Feely; 1993134 of Mar. 5, 1935 to W. W. Ford; 1997381 of Apr. 9, 1935 to Albert Horowitz; Design 95250 of Apr. 16, 1935 to N. P. Harshberger; 1999903 of Apr. 30, 1935 to N. P. Harshberger; 2000030 of May 7, 1935 to George Knapp; 2005462 of Jun. 18, 1935 to J. H. Gibson; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2013317 of Sep. 3, 1935 to B. H. Roberts and E. L. Chamberlain; 2012709 of Aug. 27, 1935 to C. P. Cowan; 2027029 of Jan. 7, 1936 to C. R. Eckert; Design 99132 of Mar. 31, 1936 to R. S. Maclean; 2035871 of Mar. 31, 1936 to C. R. Eckert; 2038102 of Apr. 21, 1936 to C. R. Eckert; 2043959 of Jun. 9, 1936 to N. P. Harshberger; 2045423 of Jun. 23, 1936 to J. A. Topping; 2047741 and 2047742 of Jul. 14, 1936 to S. P. Miller; 2056521 of Oct. 6, 1936 to J. F. Honan; 2057545 of Oct. 13, 1936 to S. C. Straub; 2059490 of Nov. 3, 1936 to Antoinette Reisig; 2059520 of Nov. 3, 1936 to N. P. Harshberger; Design 101921 of Nov. 10, 1936 to R. S. Maclean; 2063935 of Dec. 15, 1936 to Lester Kirschbraun; **Can. Pats.** 185435 of Jul. 9, 1918 to F. C. Overbury; 254365 of Oct. 6, 1925 to P. P. Welty; 283901 of Oct. 9, 1928 to Building Products Co., Ltd.; 287870 of Mar. 12, 1929 to Building Products Co., Ltd.; 304113 of Sep. 23, 1930 to C. P. Cowan; 320760 of Mar. 22, 1932 to Bird & Son, Inc.; 320864 of Mar. 22, 1932 to Patent & Licensing Corp.; 323048 of Jun. 7, 1932 to Bird & Son, Inc.; 323447 of Jun. 21, 1932 to Bird & Son, Inc.; 328694 of Dec. 20, 1933 to Patent & Licensing Corp.; 333340 of Jun. 20, 1934 to Robert Maclean; 340998 of Apr. 17, 1934 to Building Products, Ltd.; 346329 of Nov. 27, 1934 to Barrett Co.; 346637 of Dec. 11, 1934 to Barrett Co.; 347157 of Jan. 1, 1935 to Barrett Co.; 350701 and 350702 of Jun. 4, 1935 to Barrett Co.; 354872 of Dec. 24, 1935 to J. A. Feely; 363127 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.; 364944 of Mar. 23, 1937 to Certainteed Products Corp.; 365094 and 365095 of Mar. 30, 1937 to Certainteed Products Corp.

592 [p. 681] **U. S. Pats.** 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1674630 of Jun. 26, 1928 to B. C. Beckman; 1924650 of Aug. 29, 1933 to G. B. Payne; 2037507 of Apr. 14, 1936 to A. C. Fischer; **Can. Pat.** 252359 of Aug. 4, 1925 to Beckman Dawson Roofing Co.

593 [p. 681] **U. S. Pats.** 1438780 of Dec. 12, 1922 to Joseph O'Neil; 1702609 of Feb. 19, 1929 to E. W. Lesher; 2013218 of Sep. 3, 1935 to Joseph O'Neil.

594 [p. 681] **U. S. Pat.** 1473253 of Nov. 6, 1923 to R. V. Reynolds.

595 [p. 681] **U. S. Pat.** 1926257 of Sep. 12, 1933 to R. B. Bawtenheimer; 2018216 of Oct. 22, 1935 to R. S. Maclean; 2021577, 2021578 and 2021579 of Nov. 19, 1935 to L. S. Odell; **Can. Pats.** 325428 of Aug. 30, 1932 to N. L. Aberson; 332475 of May 16, 1933 to Building Products, Ltd.; 336442 of Oct. 17, 1933 to Building Products, Ltd.; 339347 of Feb. 13, 1934 to Building Products, Ltd.; 347671 of Jan. 22, 1935 to Building Products, Ltd.; 351333 and 351334 of Jul. 2, 1935 to Building Products, Ltd.

596 [p. 681] **U. S. Pat.** 2032083 of Feb. 25, 1936 to W. G. Duddleston.

597 [p. 681] **U. S. Pat.** 1840244 of Jan. 5, 1932 to J. J. Molnar.

598 [p. 681] **U. S. Pats.** 1886363 of Nov. 8, 1932 to F. C. Aufderheide; 1889091 of Nov. 29, 1932 to W. B. Fried.

599 [p. 681] **U. S. Pats.** 1926093 of Sep. 12, 1933 to S. S. Gibney; 2018805 of Oct. 29, 1935 to Antoinette Reisig; 2039536 of May 5, 1936 to W. D. Johnson.

600 [p. 682] **U. S. Pat.** 742589 of Oct. 27, 1903 to B. G. Casler.

601 [p. 682] **U. S. Pat.** 816522 of Mar. 27, 1906 to G. R. Wyman.

602 [p. 682] **U. S. Pat.** 1119553 of Dec. 1, 1914 to T. M. Vaughan; **Can. Pat.** 361274 of Oct. 20, 1936 to Crescent Brass & Pin Co.

603 [p. 682] "Protective Metallic Coatings for the Rustproofing of Iron and Steel," Circular No. 80, Bureau of Standards, Wash., D. C. (Oct. 4, 1919); "Standard Methods of Determining the Weight and Uniformity of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles" (A 90-33), A.S.T.M. Standards 1936, I, 397; "Tentative Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire" (A 191-36T), A.S.T.M. Tentative Standards 1936, 189; "Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets" (A 93-27), A.S.T.M. Stand-

ards 1936, I, 387; "Standard Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets" (A 163-36), A.S.T.M. Standards 1936, I, 448; "Tentative Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets" (A 163-35T), A.S.T.M. Tentative Standards 1936, 185; "Tentative Specifications for Electrodeposited Coatings of Zinc on Steel" (A 164-35T), A.S.T.M. Tentative Standards 1936, 192; "Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel" (A 165-35T), A.S.T.M. Tentative Standards 1936, 194.

604 [p. 682] **U. S. Pats.** 795553 of Jul. 25, 1905 to H. B. Sherman; 1435134 of Nov. 7, 1922 to Ernest Boley; **Ger. Pat.** Design 280101 of May 14, 1906 to Fritz Schmidt.

605 [p. 682] **U. S. Pats.** 1410076 of Mar. 21, 1922 to F. C. Overbury; 1940534 of Dec. 19, 1933 to Edward Caughey.

606 [p. 682] **U. S. Pat.** 2001932 of May 21, 1935 to Hamilton Maze; **Can. Pat.** 347620 of Jan. 22, 1935 to A. J. Deniston, Jr.

607 [p. 682] **U. S. Pats.** 757193 of Apr. 12, 1904 to F. S. Howard; 778863 of Jan. 3, 1905 to F. S. Howard; 1978190 of Oct. 23, 1934 to C. C. Figge; 2057003 of Oct. 13, 1936 to Clarence Bugher; **Ger. Pats.** 178525 of Jun. 22, 1905 to C. J. Carroll and J. H. Purnell; Design 387381 of Jul. 24, 1909 to Ludwig Kerkow Dachpappenfabrik.

608 [p. 683] **U. S. Pats.** 954995 of Apr. 12, 1910 to W. E. Polhemus; 1524090 of Jan. 27, 1925 to Dozier Finley; 1595079 of Aug. 10, 1926 to A. C. Fischer; 1881438 of Oct. 11, 1932 to A. C. Fischer; **Ger. Pat.** Appl. C-49370 of Jun. 29, 1934 to Aktieselskabet Jens Villadsens Fabriker.

609 [p. 683] **U. S. Pats.** 1592760 of Jul. 13, 1926 to A. C. Fischer; 1743764 of Jan. 14, 1930 to A. C. Fischer; 1769628 of Jul. 1, 1930 to A. C. Fischer; 1848076 of Mar. 1, 1932 to A. C. Fischer; 1849869 of Mar. 15, 1932 to A. C. Fischer; 1905376 of Apr. 25, 1933 to A. C. Fischer; 1983494 and 1983495 of Dec. 4, 1934 to A. C. Fischer.

610 [p. 683] **U. S. Pats.** 1241146 of Sep. 25, 1917 to R. P. Perry; 1805739 of May 19, 1931 to W. J. Moeller and A. H. Zimmerman.

611 [p. 683] "Underwriters' Laboratories Standards for Class C, Asphalt Ragfelt Sheet Roofing and Shingles," issued by the Underwriters' Laboratories, Inc., Chicago, Ill., Edition of 1936; "Federal Spec. for Roofing; Asphalt-Prepared, Smooth-Surfaced," SS-R-501, Federal Standard Stock Catalogue, Section IV, (Part 5), Aug. 1, 1933; "Federal Spec. for Roofing and Shingles; Asphalt-Prepared, Mineral-Surfaced," SS-R-521, Federal Standard Stock Catalogue, Section IV (Part 5), Aug. 1, 1933; "Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica" (D 224-35T), A.S.T.M. Tentative Standards 1936, 827; "Tentative Specs. for Asphalt Roofing Surfaced with Fine Mineral Granules" (D248-35T), A.S.T.M. Tentative Standards 1936, 823; "Tentative Specs. for Asphalt Roofing Surfaced with Coarse Mineral Granules" (D 249-35T), A.S.T.M. Tentative Standards 1936, 819.

612 [p. 683] **U. S. Pat.** 1481508 of Jan. 22, 1924 to C. A. Harris.

613 [p. 683] **U. S. Pats.** 1602314 of Oct. 5, 1926 to A. J. Signor; Reissue 18449 of May 3, 1932 to A. J. Signor; 2008952 of Jul. 23, 1935 to Erich Gach.

614 [p. 683] **U. S. Pat.** 1968281 of Jul. 31, 1934 to D. C. Cale.

615 [p. 683] **U. S. Pat.** 1925961 of Sep. 5, 1933 to N. P. Harshberger.

616 [p. 683] **U. S. Pats.** 134702 of Jan. 7, 1873 to R. U. Piper; 348844 of Sep. 7, 1886 to David Harger; 1109738 of Sep. 8, 1914 to W. P. Coldren; 1263218 and 1263219 of Apr. 16, 1918 to A. C. Fischer; 1332223 of Mar. 2, 1920 to J. R. McCord, Jr.; 1334178 of Mar. 16, 1920 to W. B. Sharp; 1446455 of Feb. 27, 1923 to A. C. Fischer; 1447986 of Mar. 13, 1923 to W. C. Johnson; 1480246 of Jan. 8, 1924 to C. E. Douglas; 1600667 of Sep. 21, 1926 to A. C. Fischer; 1626802 of May 3, 1927 to A. C. Fischer; **Ger. Pats.** 188146 of Feb. 4, 1905 to Roland Risse; 286903 of Sep. 20, 1913 to Ludwig Schwabe.

617 [p. 684] **U. S. Pats.** 255087 of Mar. 14, 1882 to L. L. Sagendorph; 257616 of May 9, 1882 to F. E. Sagendorph; 259228 of Jun. 6, 1882 to O. A. Smith and F. L. Kane; 291440 of Jan. 1, 1884 to C. M. Warren; 362246 of May 3, 1887 to J. D. Brown, Jr.; 362732 of May 10, 1887 to W. H. Fay; 754273 of Mar. 8, 1904 to W. H. Bache; 876843 of Jan. 14, 1908 to A. E. Roever; 936139 of Oct. 5, 1909 to Charles Lagergren; 954995 of Apr. 12, 1910 to W. E.

Polhemus; 984860 of Feb. 21, 1911 to F. E. Smith; 1087321 of Feb. 17, 1914 to Calvin Russell; 1276632 of Aug. 20, 1918 to Ernest Flagg; 1356930 of Oct. 26, 1920 to J. R. McCord, Jr.; 1471156 of Oct. 16, 1923 to A. A. Griswold; 1612718 of Dec. 28, 1926 to J. F. Grice; 1618902 of Feb. 22, 1927 to Henry Yost; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1684179 of Sep. 11, 1928 to A. A. Griswold; 1747492 of Feb. 18, 1930 to J. S. Skoglund and C. M. Engebretson; 1755538 of Apr. 22, 1930 to A. H. Draughon, Jr.; 1794072 of Feb. 24, 1931 to A. A. Griswold; 1810808 of Jun. 16, 1931 to Henry Yost; 1825732 of Oct. 6, 1931 to G. A. Holman; 1952383 of Mar. 27, 1934 to F. C. McClanahan; 1978237 of Oct. 23, 1934 to O. P. Valiquet and J. F. Chandler; 1990776 of Feb. 12, 1935 to C. J. Dexter; 2060739 of Nov. 10, 1936 to Homer Maddux; **Can. Pats.** 234217 of Sep. 18, 1923 to H. P. Baltzer; 278134 of Feb. 28, 1928 to Henry Yost; 329550 of Jan. 24, 1933 to G. A. Holman; 351115 of Jun. 25, 1935 to O. P. Valiquet; **Ger. Pats.** 188146 of Feb. 4, 1905 to Roland Risse; 189198 of Sep. 19, 1905 to Julius Fichtel; Design 387381 of Jul. 24, 1909 to Ludwig Kerkow Dachpappenfabrik; Design 1269843 of Jul. 7, 1933 to Hans Meier.

618 [p. 684] **U. S. Pats.** 887532 of May 12, 1908 to H. B. Sherman; 967208 of Aug. 16, 1910 to J. F. Leslie; 1321958 of Nov. 18, 1919 to H. R. Wardell; **Can. Pat.** 139036 of Mar. 12, 1912 to H. W. Johns-Manville Co.

619 [p. 684] **U. S. Pats.** 973902 of Oct. 25, 1910 to W. H. Woerheide; 981362 of Jan. 10, 1911 to J. H. Bell; 985501 of Feb. 28, 1911 to J. H. Bell; 1017611 of Feb. 13, 1912 to H. R. Wardell; 1018946 of Feb. 27, 1912 to H. W. Topping; 1055848 of Mar. 11, 1913 to W. H. Woerheide; 1066959 of Jul. 8, 1913 to J. A. Topping; 1076639 of Oct. 21, 1913 to W. H. Nicholls and A. C. Carmichael; 1101896 of Jun. 30, 1914 to W. H. Woerheide; 1109738 of Sep. 8, 1914 to W. P. Coldress; 1148260 of Jul. 27, 1915 to P. W. Stansbury; 1148647 of Aug. 3, 1915 to E. J. Yetter; 1150261 of Aug. 17, 1915 to A. C. Fischer; 1163269 of Dec. 7, 1915 to H. B. Sherman; 1186257 of Jun. 6, 1916 to W. H. Woerheide; 1187532 of Jun. 20, 1916 to H. C. Kettelson; 1273500 of Jul. 23, 1918 to H. C. Kettelson; 1348858 of Aug. 10, 1920 to L. M. Ford; 1357265 of Nov. 2, 1920 to W. H. Woerheide; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1657851 of Jan. 31, 1928 to D. R. Bard; 1692401 of Nov. 20, 1928 to D. R. Bard; 1761599 of Jun. 3, 1930 to J. A. Topping; 1930632 of Oct. 17, 1933 to J. A. Topping; **Can. Pats.** 284462 of Nov. 6, 1928 to D. R. Bard; 335689 of Sep. 12, 1933 to J. A. Topping; 354586 of Dec. 3, 1935 to Galt Art Metal Co.; **Brit. Pat.** 162043 of Apr. 18, 1921 to Evelyn Hurden, A. P. Hurden and S. E. Beeson; **Ger. Pats.** 139645 of Jun. 8, 1902 to Anton Köstner; 188146 of Feb. 4, 1905 to Roland Risse; Design 402505 of Nov. 2, 1909 to Willibald Elsel; Design 474583 of Jul. 10, 1911 to Fritz Sonnemann; 275332 of Aug. 19, 1911 to J. T. Neuhaus.

620 [p. 684] **U. S. Pats.** 757193 of Apr. 12, 1904 to F. S. Howard; 778863 of Jan. 3, 1905 to F. S. Howard; 1052258 of Feb. 4, 1913 to E. J. Kenney; 1225972 of May 15, 1917 to H. C. Kettelson; 1237270 of Aug. 21, 1917 to Herbert Abraham; 1242675 of Oct. 9, 1917 to S. M. Ford; 1297323 of Mar. 18, 1919 to R. C. Clark; 14711156 of Oct. 16, 1923 to A. A. Griswold; 1479067 of Jan. 1, 1924 to A. A. Griswold; **Can. Pat.** 97905 of Mar. 6, 1906 to F. S. Howard.

621 [p. 684] **U. S. Pats.** 60708 of Jan. 1, 1867 to C. J. Fay; 61184 of Jan. 15, 1867 to L. D. Ford; 154843 of Sep. 8, 1874 to Rowell Colby; 223671 of Jan. 20, 1880 to Thomas Fugate; 257616 of May 9, 1882 to F. E. Sagendorph; 259228 of Jun. 6, 1882 to O. A. Smith and F. L. Kane; 285131 of Sep. 18, 1883 to J. F. Hoffman; 322153 of Jul. 14, 1885 to L. F. Blair and J. W. Roche; 332570 of Dec. 15, 1885 to W. H. Stewart; 352619 of Nov. 16, 1886 to F. L. Kane; 354311 of Dec. 14, 1886 to C. A. Favel; 362246 of May 3, 1887 to J. D. Brown, Jr.; 362732 of May 10, 1887 to W. H. Fay; 632825 of Sep. 12, 1899 to R. J. Redick; 636022 of Oct. 31, 1899 to G. D. Crabbs and W. H. Pendery; 652150 of Jun. 19, 1900 to F. W. Terpenning; 669315 of Mar. 5, 1901 to W. P. Whitmore; 713588 of Nov. 18, 1902 to John Ayrault, Jr.; 754273 of Mar. 8, 1904 to W. H. Bache; 762220 of Jun. 7, 1904 to Jordan Williams (metal); 813163 of Feb. 20, 1906 to W. J. Moeller; 821606 of May 29, 1906 to E. N. Brogan; 835899 of Nov. 13, 1906 to W. J. Moeller; 855757 of Jun. 4, 1907 to G. D. Crabbs and W. H. Pendery; 868930 of Oct. 22, 1907 to A. E. Kirk; 876843 of Jan. 14, 1908 to A. E. Roever; 936139 of Oct. 5, 1909 to Charles Lagergren; 948116 of Feb. 1, 1910 to W. H. Pendery; 972957 of Oct. 18, 1910 to H. R. Wardell; 984860 of Feb. 21, 1911 to F. E. Smith; 1018524 of Feb. 27, 1912 to J. J. Smiley; 1083243 of Dec. 30, 1913 to W. C. Edwards, Jr.; 1087321 of Feb. 17, 1914 to

Calvin Russell; 1100955 of Jun. 23, 1914 to E. B. Coburn; 1158266 of Oct. 26, 1915 to F. C. Overbury and H. C. Platts; 1338262 of Apr. 27, 1920 to C. A. Statler; 1362755 of Dec. 21, 1920 to C. A. Statler; 1372208 of Mar. 22, 1921 to C. A. Statler; 1434200 of Oct. 31, 1922 to W. M. Chase; 1435623 of Nov. 14, 1922 to I. C. Saxe; 1447986 of Mar. 13, 1923 to W. C. Johnson; 1489898 of Apr. 8, 1924 to Svend Petersen; 1552883 of Sep. 8, 1925 to Richard Rudel; 1568227 of Jan. 5, 1926 to F. P. Leonard; 1600667 of Sep. 1, 1926 to A. C. Fischer; 1655494 of Jan. 10, 1928 to C. P. Cowan; 1684179 of Sep. 11, 1928 to A A. Griswold; 1690792 of Nov. 6, 1928 to R. C. Neptune; 1710104 of Apr. 23, 1929 to R. C. Neptune; 1723927 of Aug. 6, 1929 to A. C. Fischer; 1829488 of Oct. 27, 1931 to E. C. Miller; 1873944 of Aug. 23, 1932 to J. E. Black; 1882177 of Oct. 11, 1932 to L. W. Burris; 1950840 of Mar. 13, 1934 to J. E. Cook; 2003503 of Jun. 4, 1935 to S. L. Eason; 2003728 of Jun. 4, 1935 to Hermann von Forster and Samuel de Lange; 2013330 of Sep. 3, 1935 to L. M. Abraham; **Can. Pats.** 29795 of Sep. 1, 1885 to W. H. Fay; 84226 of Dec. 8, 1903 to John Ayrault; 98890 and 98891 of May 8, 1906 to Philip Carey Mfg. Co.; 107146 of Aug. 27, 1907 to E. N. Brogan; 117009 of Mar. 2, 1909 to A. E. Kirk; 145536 of Jan. 28, 1913 to Calvin Russell; 218121 of May 2, 1922 to Fred Fredericksen; 247225 of Mar. 3, 1925 to Svend Petersen; 259005 of Mar. 16, 1926 to Bird & Son, Ltd.; 260351 of May 4, 1926 to J. A. Blair; 364370 of Feb. 23, 1937 to S. L. Eason; **Ger. Pats.** 53010 of Dec. 29, 1889 to Adalbert Kelm; 119104 of May 2, 1900 to Paul Fuhrmann; 574880 of Aug. 31, 1929 to Waclaw Szpakowski; 638035 of Jun. 30, 1934 to Aktieselskabet Jens Villardsens; Appl. W-95428 of Nov. 17, 1934 to Fritz Werner; **Austrian Pat.** 65252 of Dec. 15, 1913 to Heinrich Schwarz.

622 [p. 684] **U. S. Pats.** 61899 of Feb. 5, 1867 to I. C. Wands; 97796 of Dec. 14, 1869 to H. G. Noble; 143471 of Oct. 7, 1873 to R. A. Smith; 195593 of Sep. 25, 1877 to H. F. Evans; 511385, 511386 and 511387 of Dec. 26, 1893 to James Smith; 674579 of May 21, 1901 to W. H. Grow; 993686 of May 30, 1911 to F. S. Howard; 1051702 of Jan. 28, 1913 to H. W. Deming; 1061685 of May 13, 1913 to George Newton; 1126114 of Jan. 26, 1915 to A. S. Spiegel; 1137895 of May 4, 1915 to A. W. Phippen; 1144265 of Jun. 22, 1915 to Herman von Uffel; 1147582 of Jul. 20, 1915 to Herman von Uffel; 1158267 and 1158268 of Oct. 26, 1915 to F. C. Overbury and H. C. Platts; 1210855 of Jan. 2, 1917 to R. L. Shainwald, Jr.; 1221370 of Apr. 3, 1917 to F. C. Overbury and H. C. Platts; 1247743 of Nov. 27, 1917 to W. D. Thompson; 1421040 of Jun. 27, 1922 to G. E. Swenson; 1447175 of Mar. 6, 1923 to O. L. Henderson; 1517944 of Dec. 2, 1924 to J. F. Bobbitt; 1544866 of Jul. 7, 1925 to G. F. Slater; 1808286 of Jun. 2, 1931 to G. G. Britton; 1908127 of May 9, 1933 to W. T. Deacon; **Ger. Pats.** 24612 of Oct. 5, 1882 to David Röhm; 44463 of Dec. 24, 1887 to Julius Wascher; 51585 of Jun. 5, 1889 to Ludwig Hatschek; Design 402505 of Nov. 2, 1909 to Willibald Elsel; 275332 of Aug. 19, 1911 to J. T. Neuhaus; 292077 of Jan. 20, 1915 to Hans Christen; Design 783579 of Mar. 26, 1920 to Otto Faust; 574880 of Aug. 31, 1929 to Waclaw Szpakowski.

623 [p. 684] **U. S. Pat.** 1986471 of Jan. 1, 1935 to A. C. Fischer.

624 [p. 684] **U. S. Pats.** 1901356 of Mar. 14, 1933 to J. H. Plunkett; 2023030 of Dec. 3, 1935 to J. H. Plunkett; **Can. Pats.** 342629 of Jun. 26, 1934 to Patent & Licensing Corp.; 362410 of Dec. 8, 1936 to Patent & Licensing Corp.

625 [p. 684] **U. S. Pat.** 1008435 of Nov. 14, 1911 to H. M. Reynolds.

626 [p. 684] **U. S. Pat.** 694304 of Feb. 25, 1902 to C. S. Bird and J. B. Hanscom.

627 [p. 684] **Can. Pat.** 354392 of Nov. 26, 1935 to Building Products, Ltd.

628 [p. 684] **U. S. Pat.** 803713 of Nov. 7, 1905 to H. M. Reynolds.

629 [p. 684] **U. S. Pat.** 852397 of Apr. 30, 1907 to W. L. Penney.

630 [p. 684] **U. S. Pat.** 713938 of Nov. 18, 1902 to W. H. Bache.

631 [p. 684] **U. S. Pat.** 873046 of Dec. 10, 1907 to F. S. Howard.

632 [p. 684] **U. S. Pats.** 742558 of Oct. 27, 1903 to W. H. Bache; 874160 of Dec. 17, 1907 to Purlan Buckborough.

633 [p. 684] **U. S. Pats.** 825239 of Jul. 3, 1906 to M. C. Ohnemus; 919607, 919608 and 919739 of Apr. 27, 1909 to G. W. Loughman; 923362 of Jun. 1, 1909 to G. W. Loughman; 1029947 of Jun. 18, 1912 to C. J. Rothermel; 1517940 of Dec. 2, 1924 to J. W. Bellairs; 1548632 of Aug. 4, 1925 to C. J. Rothermel; 1912388 of Jun. 6, 1933 to Samuel Slovin; **Can. Pat.** 99185 of May 22, 1906 to M. C. Ohnemus.

634 [p. 684] **U. S. Pats.** 996510 and 996511 of Jun. 27, 1911 to G. J. Oltsch; 1298811 of Apr. 1, 1919 to A. S. Spiegel; 1781016 of Nov. 11, 1930 to Henry Kielberg; **Can. Pat.** 207018 of Dec. 28, 1920 to Roofing Patents Co.

635 [p. 684] **U. S. Pats.** 1312202 of Aug. 5, 1919 to R. P. Perry; 1312211 of Aug. 5, 1919 to C. J. Rothermel.

636 [p. 684] **U. S. Pats.** 980406 of Jan. 3, 1911 to B. G. Casler; 1543052 of Jun. 23, 1925 to A. W. Brown.

637 [p. 684] **U. S. Pat.** 1651664 of Dec. 6, 1927 to C. E. Bowers.

638 [p. 684] **U. S. Pat.** 1572140 of Feb. 9, 1926 to O. A. Heppes; **Can. Pat.** 143374 of Oct. 15, 1912 to C. W. Dohm.

639 [p. 685] **U. S. Pat.** 1380475 of Jun. 7, 1921 to O. A. Heppes.

640 [p. 685] **U. S. Pat.** 1365403 of Jan. 11, 1921 to R. F. Holway.

641 [p. 685] **U. S. Pats.** 1312202 of Aug. 5, 1919 to R. P. Perry; 1312211 of Aug. 5, 1919 to C. J. Rothermel.

642 [p. 685] **U. S. Pats.** 1359569 of Nov. 23, 1920 to C. S. Bird; 1429836 of Sep. 19, 1922 to C. S. Bird; 1765839 of Jun. 24, 1930 to Lester Kirschbraun; Design 102555 of Dec. 29, 1936 to H. W. Holland.

643 [p. 685] **U. S. Pat.** 1873887 of Aug. 23, 1932 to O. A. Heppes.

644 [p. 685] **U. S. Pats.** 1324039 of Dec. 9, 1919 to C. F. Evans; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1482764 of Feb. 5, 1924 to George Ritter; 1493616 of May 13, 1924 to J. E. Domagall; 1686513 of Oct. 9, 1928 to W. H. Cady.

645 [p. 685] **U. S. Pats.** 1604182 and 1604183 of Oct. 26, 1926 to J. P. Markert.

646 [p. 685] **Can. Pat.** 140335 of May 14, 1912 to E. H. Cunningham.

647 [p. 685] **Ger. Pat.** Design 1250165 of Jan. 9, 1933 to Nikolaus Jungblut.

648 [p. 685] **U. S. Pat.** 840103 of Jan. 1, 1907 to R. W. Bird; **Ger. Pat.** Appl. Sch-111.30 of Dec. 24, 1930 to Hans Schmitt.

649 [p. 685] **U. S. Pats.** 372316 of Nov. 1, 1887 to C. B. Hutchins; 761138 of May 31, 1904 to C. S. Bird and P. R. Allen; 845414 of Feb. 26, 1907 to Samuel Herbert.

650 [p. 685] **U. S. Pat.** 1928751 of Oct. 3, 1933 to T. S. Bradley.

651 [p. 685] **U. S. Pat.** 1166541 of Jan. 4, 1916 to F. C. Overbury.

652 [p. 685] **Can. Pat.** 254431 of Oct. 6, 1925.to Paraffine Cos., Inc.

653 [p. 685] **U. S. Pats.** 1418377 of Jun. 6, 1922 to C. B. Jaynes; 1467017 of Sep. 4, 1923 to G. C. Trice.

654 [p. 686] **U. S. Pats.** 18186 of Sep. 15, 1857 to W. H. Carver and J. Beckley; 40542 of Nov. 3, 1863 to L. S. Mills and C. H. Smith; 55891 of Jun. 26, 1866 to Rufus Norwood; 58246 of Sep. 25, 1866 to William Gilbert; 61787 of Feb. 5, 1867 to J. R. Woodworth; 61878 of Feb. 5, 1867 to John Scanlan; 63429 of Apr. 2, 1867 to F. O. Rogers; 64493 of May 7, 1867 to Marvin Converse and A. C. Torry; 74430 of Feb. 11, 1868 to Leander Rodney; 76375 of Apr. 7, 1868 to Edward Atkinson; 82507 of Sep. 29, 1868 to T. E. Wood; 93304 of Aug. 3, 1869 to C. B. Hutchins; 94493 of Sep. 7, 1869 to Benjamin Hinkley; 101071 of Mar. 22, 1870 to T. E. Wood; 102061 of Apr. 19, 1870 to W. M. Stuart; 124192 of Mar. 5, 1872 to D. W. Bailey; 136516 of Mar. 4, 1873 to J. H. Hood; 140945 of Jul. 15, 1873 to Charles Mueller; 146608 of Jan. 20, 1874 to Tobias New; 147962 of Feb. 24, 1874 to Tobias New; 156537 of Nov. 3, 1874 to Edward Churchill; 156639 of Nov. 10, 1874 to Jeduthan Kittredge; 161762 of Apr. 6, 1875 to Allan Cummings; 179131 of Jun. 27, 1876 to Lewis Peirce; 197170 of Nov. 13, 1877 to Lewis Peirce; 199210 of Jan. 15, 1878 to F. L. Kane; 200122 of Feb. 12, 1878 to M. B. Bailey; 202493 of Apr. 16, 1878 to E. B. Warren; 205132 of Jun. 18, 1878 to Lewis Peirce; Reissue 8414 of Sep. 10, 1878 to Tobias New; 209705 of Nov. 5, 1878 to Tobias New; 209830 of Nov. 12, 1878 to Tobias New; 227682 of May 18, 1880 to S. L. Foster and W. H. Benton; Reissue 9314 of Jul. 20, 1880 to S. L. Foster and W. H. Benton; 236386 of Jan. 4, 1881 to C. M. Warren; 270943 of Jan. 23, 1883 to S. L. Foster; 296163 of Apr. 1, 1884 to Levi Haas; and Dennis Howarth; 313971 of Mar. 17, 1885 to T. H. White; 438324 of Oct. 14, 1890 to M. W. Powell; 677058 of Jun. 25, 1901 to Emil Borgeson and Axel Wennerberg; 712193 of Oct. 28, 1902 to F. L. Kane; 720811 of Feb. 17, 1903 to John Ingram; 798131 of Aug. 29, 1905 to F. W. Gezelschap and Arthur Winding; 805745 and 805746 of Nov. 28, 1905

to F. N. Pease; 822602 of Jun. 5, 1906 to August Gross and A. C. Horn; 842079 of Jan. 22, 1907 to E. B. Campbell; 846572 of Mar. 12, 1907 to C. J. Kunzler; 879372 of Feb. 18, 1908 to C. C. Dill; 921538 of May 11, 1909 to John Glassford; 1012903 of Dec. 26, 1911 to Henry Olmsted, Jr.; 1044773 and 1044774 of Nov. 19, 1912 to Harry Gillett; 1110330 of Sep. 15, 1914 to F. J. McClaskey; 1170521 of Feb. 8, 1916 to S. B. Faison; 1207726 of Dec. 12, 1916 to C. V. Eades; 1230396 of Jun. 19, 1917 to F. L. Foster; 1235270 of Jul. 31, 1917 to J. B. Wise; 1261276 of Apr. 2, 1918 to J. C. Norton; 1284997 of Nov. 19, 1918 to O. A. Bigler; 1343478 of Jun. 15, 1920 to H. H. Robertson; 1363539 of Dec. 28, 1920 to J. O. Boylan; 1422526 of Jul. 11, 1922 to J. M. Berdan; 1606496 of Nov. 9, 1926 to C. J. Beckwith and R. K. Austin; 1705160 of Mar. 12, 1929 to J. P. Stagg; 1780622 of Nov. 4, 1930 to W. H. Lawrence; 1880429 of Oct. 4, 1932 to F. H. Ford; 1958871 of May 15, 1934 to E. M. Tucker; 1967105 of Jul. 17, 1934 to H. L. Seymour; 1994262 of Mar. 12, 1935 to Arthur Winding; 2007403 of Jul. 9, 1935 to H. C. Macan; **Can. Pats.** 7669 of Jul. 17, 1877 to Alexander McLean; 15035 of Jun. 28, 1882 to J. W. Paterson; 28896 of Apr. 15, 1888 to Clinton French; 216492 of Mar. 7, 1922 to Johns-Manville, Inc.; 278755 of Mar. 20, 1928 to Johns-Manville Corp.; 284700 of Nov. 13, 1928 to W. A. Moffatt; 347154 of Jan. 1, 1935 to Barrett Co.; 360554 of Sep. 15, 1936 to Anaconda Copper Mining Co.; **Brit. Pats.** of 1897 (Nov. 26), 27884 to Robert Glendenning; of 1898 (Dec. 23), 27118 to J. J. Ingham; 427851 of Jul. 13, 1934 to Jacques Gevers; 431351 of Jan. 5, 1934 to W. B. Ranson; **Ger. Pats.** 212527 of Oct. 30, 1906 to Ludwig Esselborn; Design 325524 of Sep. 11, 1907 to Fritz Grimm; Design 330871 of Feb. 28, 1907 to Wirtschaftliche Vereinigung für die Westdeutsche Dachpappenindustrie, G.m.b.H.; Design 336320 of Mar. 9, 1908 to B. Lohse & Rothe; Design 347273 of Mar. 20, 1907 to Büsscher & Hoffmann, G.m.b.H.; 219448 of Mar. 17, 1908 to Friedrich Drexler; Design 414575 of Sep. 3, 1909 to Albin Karg; Design 543093 of Apr. 16, 1912 to Ernst Zorn; Appl. G-108.30 of Jan. 16, 1928 to C. Gartenmann & Cie.; Appl. H-137428 of Sep. 9, 1933 to Heinrich Habig.

655 [p. 686] **U. S. Pats.** 61746 of Feb. 5, 1867 to R. O. Lowrey; 133247 of Nov. 19, 1872 to John Park; 147962 of Feb. 24, 1874 to Tobias New; Reissue 8414 of Sep. 10, 1878 to Tobias New; 209131 of Oct. 22, 1878 to Tobias New; 217916 and 217917 of Jul. 29, 1879 to C. M. Warren; 227682 of May 18, 1880 to S. L. Foster and W. H. Benton; Reissue 9314 of Jul. 20, 1880 to S. L. Foster and W. H. Benton; 318023 of May 19, 1885 to M. W. Powell; 712193 of Oct. 28, 1902 to F. L. Kane; 712308 of Oct. 28, 1902 to F. L. Kane; 2019647 of Nov. 5, 1935 to Frederick Basten and Evelyn Hurden; 2031249 of Feb. 18, 1936 to O. S. Bowman; **Brit. Pat.** of 1890 (Apr. 5), 2619 to Johann Schultz and Edward Hoff; **Ger. Pats.** 24612 of Oct. 5, 1882 to David Röhm; Design 419504 of Oct. 12, 1909 to Hilarius Knobel; Design 441025 of Mar. 14, 1910 to Hilarius Knobel.

656 [p. 686] "Federal Spec. for Roofing; Built-up, Type 5TWS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-578; Bureau of Standards Circular C-179.

657 [p. 686] "Federal Spec. for Roofing; Built-up, Type 5AWS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-572; Bureau of Standards Circular C-171.

658 [p. 686] "Federal Spec. for Roofing; Built-up, Type 5TCS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-575; Bureau of Standards Circular C-177.

659 [p. 686] "Federal Spec. for Roofing; Built-up, Type 5ACS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-569; Bureau of Standards Circular C-174.

660 [p. 686] "Federal Spec. for Roofing; Built-up, Type 4TWS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-566; Bureau of Standards Circular C-178.

661 [p. 686] "Federal Spec. for Roofing; Built-up, Type 4AWS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-560; Bureau of Standards Circular C-170.

662 [p. 686] "Federal Spec. for Roofing; Built-up, Type 4ACS (Construction of),"

Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-557; Bureau of Standards Circular C-173.

663 [p. 686] "Federal Spec. for Roofing; Built-up, Type 4TCS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-563; Bureau of Standards Circular C-176.

664 [p. 686] "Federal Spec. for Roofing; Built-up, Type 3TCS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-554; Bureau of Standards Circular C-175.

665 [p. 686] "Federal Spec. for Roofing; Built-up, Type 3ACS (Construction of)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-551; Bureau of Standards Circular C-172.

666 [p. 686] "Federal Spec. for Flashings; Metal (Installation with Bituminous Built-up Roofing)," Federal Standard Stock Catalogue, Section IV, (Part 5), QQ-F-451; Bureau of Standards Circular C-180; "Federal Spec. for Flashings; Plastic (Installation with Bituminous Built-up Roofing)," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-F-451; Bureau of Standards Circular C-181.

667 [p. 686] **U. S. Pats.** 34653 of Mar. 11, 1862 to Zoadk Street; 40542 of Nov. 3, 1863 to L. S. Miller; 94689 of Sep. 7, 1869 to R. K. Kille; Reissue 3701 of Nov. 2, 1869 to R. K. Kille; 141437 of Aug. 5, 1873 to A. G. Hennion; 145705 of Dec. 16, 1873 to Horace Wheeler; 313971 of Mar. 17, 1885 to T. H. White; 358994 of Mar. 8, 1887 to W. H. Stewart; 359925 of Mar. 22, 1887 to Clinton French; 756180 of Mar. 29, 1904 to J. H. Munro; 1728795 of Sep. 17, 1929 to J. H. Griffin; 1930427 of Oct. 10, 1933 to J. H. Griffin; **Can. Pat.** 42242 of Mar. 10, 1893 to R. A. Chesebrough.

668 [p. 686] **U. S. Pats.** 137566 of Apr. 8, 1873 to E. R. Percy; 438973 of Oct. 21, 1890 to J. C. Zallée; **Can. Pat.** 337819 of Dec. 12, 1933 to Frazzi, Ltd.; **Brit. Pat.** 389668 of Apr. 28, 1932 to Laurent Noesen; **Ger. Pats.** 212527 of Oct. 30, 1906 to Ludwig Esselborn; 580391 of Jun. 17, 1930 to Karl Meier; 594470 of Jan. 17, 1928 to Richard Franz and Karl Meier.

669 [p. 686] "Federal Spec. for Surfacing-Material; (for) Bituminous Built-up Roofing," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-S-791; Bureau of Standards Circular C-158 (Canceled Aug. 1, 1923).

670 [p. 687] **U. S. Pat.** 1977669 of Oct. 23, 1934 to T. M. Dantz.

671 [p. 688] **U. S. Pats.** 1427755 of Aug. 29, 1922 to W. A. Harris; 2061066 of Nov. 17, 1936 to C. R. Eckert; **Can. Pats.** 346328 of Nov. 27, 1934 to Barrett Co.; 362228 of Dec. 1, 1936 to Barrett Co.

672 [p. 688] **U. S. Pat.** 1340347 of May 18, 1920 to C. E. Rahr.

673 [p. 688] **U. S. Pats.** 1493447 of May 6, 1924 to O. A. Heppes; 1788121 of Jan. 6, 1931 to F. C. Overbury; **Can. Pat.** 249096 of Apr. 28, 1925 to Flintkote Co.; **Ger. Pat.** Design 326523 of Nov. 30, 1907 to Wilhelm Zerlin.

674 [p. 688] **U. S. Pat.** 1441861 of Jan. 9, 1923 to T. B. Lehon.

675 [p. 688] **U. S. Pats.** 1752827 of Apr. 1, 1930 to W. J. Young; 1904080 of Apr. 18, 1933 to H. L. Plummer and D. L. Irwin; 1938496 of Dec. 5, 1933 to H. L. Plummer and D. L. Irwin; 1965972 of Jul. 10, 1934 to H. R. Balph; **Can. Pats.** 316012 of Oct. 13, 1931 to H. L. Plummer and D. L. Irwin; 335007 of Aug. 22, 1933 to H. L. Plummer and D. L. Irwin; **Brit. Pat.** 337525 of Sep. 5, 1929 to H. L. Plummer and D. L. Irwin; **Ger. Pats.** 552785 of May 18, 1930 to H. L. Plummer and D. L. Irwin; Design Pat. 1375002 of Apr. 29, 1936 to Theodor Greis.

676 [p. 688] **U. S. Pats.** 1403510 of Jan. 17, 1922 to J. F. Loucks; 1974134 of Feb. 13, 1934 to G. W. Mills, Jr.

677 [p. 688] **U. S. Pats.** 1541130 of Jun. 9, 1925 to Dozier Finley; 1894754 of Jan. 17, 1933 to M. E. Campbell; 2048663 of Jul. 21, 1936 to S. P. Miller; **Ger. Pat.** Appl. L-78186 of Mar. 18, 1931 to Johannes Lotzin.

678 [p. 688] "Asphalt Shingle and Roofing Institute, New York, May, 1928.

679 [p. 688] **U. S. Pats.** 1467017 of Sep. 4, 1923 to G. C. Trice; 1636095 of Jul. 19, 1927 to H. G. Hose and W. E. Ramsdell; **Can. Pat.** 362706 of Dec. 15, 1936 to F. C. Overbury.

680 [p. 688] **U. S. Pats.** 1601731 of Oct. 5, 1926 to John Flood; 18454164 of Apr. 12, 1932 to A. C. Fischer; 1923002 of Aug. 15, 1933 to R. T. Johnston; **Swiss Pat.** 10454 of Jun. 1, 1895 to Gebrüder Braun.

681 [p. 688] **U. S. Pats.** 1560608 of Nov. 10, 1925 to R. R. Richardson; 1584887 of May 18, 1926 to Gustaf Nyholm.

682 [p. 688] **U. S. Pat.** 1685559 of Sep. 25, 1928 to W. R. Tobias.

683 [p. 688] **U. S. Pats.** 1668269 of May 1, 1928 to S. B. Faison; 1917930 of Jul. 11, 1933 to A. C. Fischer.

684 [p. 688] **U. S. Pat.** 1890928 of Dec. 13, 1932 to E. R. Black.

685 [p. 688] **U. S. Pat.** 1810674 of Jun. 16, 1931 to H. G. Muir.

686 [p. 688] List of Inspected Mechanical Appliances, issued by the Underwriters' Laboratories, Inc., Chicago, Jan. 1936.

687 [p. 693] "Fire Tests of Wood Shingles and Asphalt Prepared Roofings," by S. H. Ingberg, N. D. Mitchell and H. E. Newcomer, Preliminary Report Prepared by the Bureau of Standards, Washington, D. C, Mar., 1926; also "Preliminary Draft of an Investigation of Bituminous Built-up Roofing," by E. H. Berger, Bureau of Standards, Washington, D. C., Apr. 7, 1922.

688 [p. 693] "Report of the Technical Committee on the Combustibility of Roofing Materials," Dominion Fire-Prevention Association, Ottawa, Canada, Mar. 31, 1926.

689 [p. 693] **Fr. Pat.** 610916 of Nov. 23, 1925 to Auguste Lannoye.

690 [p. 693] **U. S. Pats.** 1998779, 1998780 and 1998781 of Apr. 23, 1935 to R. G. Jackson.

691 [p. 694] **U. S. Pat.** 1528436 of Mar. 3, 1925 to Lester Kirschbraun.

692 [p. 694] **U. S. Pat.** 1549991 of Aug. 18, 1925 to Lester Kirschbraun.

693 [p. 694] **U. S. Pat.** 1417839 of May 30, 1922 to Lester Kirschbraun; **Ger. Pat.** 401546 of Jun. 2, 1921 to Lester Kirschbraun.

694 [p. 694] **U. S. Pat.** 1720780 of Jul. 16, 1929 to C. S. Cooley.

695 [p. 694] **U. S. Pat.** 1297009 of Mar. 11, 1919 to George Prifold.

696 [p. 694] **U. S. Pat.** 1273536 of Jul. 23, 1918 to George Prifold.

697 [p. 694] **U. S. Pats.** 1606735 of Nov. 9, 1926 to E. M. Lines and J. F. Maguire; 1618102 of Feb. 15, 1927 to J. C. Morrell; 1948959 of Feb. 27, 1934 to M. Croce; **Can. Pat.** 268699 of Mar. 1, 1927 to Bird & Son, Inc.

698 [p. 694] **U. S. Pats.** 1176853 of Mar. 28, 1916 to George Prifold; 1184584 of May 23, 1916 to W. D. Snow; **Brit. Pat.** of 1913 (Feb. 14), 3841 to E. C. R. Marks; **Ger. Pat.** Appl. B-72226 of Feb. 13, 1913 to Barrett Mfg. Co. (Rejected).

699 [p. 694] **U. S. Pat.** 1008296 of Nov. 7, 1911 to Judd Smith.

700 [p. 694] **Can. Pat.** 313437 of Jul. 21, 1931 to Armstrong Cork Co.; **Brit. Pats.** 348892 of Jun. 22, 1929 to Armstrong Cork Co.; 349792 of Jun. 12, 1930 to Armstrong Cork Co.

701 [p. 694] **Brit. Pat.** 184953 of Aug. 31, 1922 to C. R. Leech.

702 [p. 694] **U. S. Pats.** 1131317 of Mar. 9, 1915 to C. S. Bird; 1956197 of Apr. 24, 1934 to E. M. Lines.

703 [p. 694] **U. S. Pat.** 1442012 of Jan. 9, 1923 to W. E. Summer.

704 [p. 694] **U. S. Pats.** 1637762 of Aug. 2, 1927 to J. B. Campbell; 1823238 of Sep. 15, 1931 to J. B. Campbell; **Can. Pat.** 282863 of Aug. 28, 1928 to Carthage Mills, Inc.; **Brit. Pats.** 372031 of Mar. 17, 1931 to Reginald Brown; 372962 of May 19, 1932 to Rylands & Sons, Ltd.; 440680 of Nov. 6, 1934 to Norman Royle.

705 [p. 694] **U. S. Pats.** 1765748 of Jun. 24, 1930 to M. C. Teague; 2011149 and 2011150 of Aug. 13, 1935 to R. G. Jackson; 2056958 of Oct. 13, 1936 to E. T. A. Coughlin; Reissue 20182 of Nov. 24, 1936 to R. G. Jackson; **Can. Pat.** 356170 of Feb. 25, 1936 to Sandura Co., Inc.; **Brit. Pats.** 376033 of Jul. 21, 1931 to Flintkote Co.; 415049 of Aug. 13, 1934 to Allon Skipsey.

706 [p. 694] **U. S. Pat.** 1784810 of Dec. 16, 1930 to R. D. Bonney and W. Y. Irwin, Jr.

707 [p. 695] **U. S. Pat.** 1868038 of Jul. 19, 1932 to W. E. Whitney and H. G. Hassenplug.

708 [p. 695] **Brit. Pat.** 330384 of May 16, 1929 to John Lunan.

709 [p. 695] **U. S. Pats.** 1802009 of Apr. 21, 1931 to Dozier Finley; 2011149 and 2011150

of Aug. 13, 1935 to R. G. Jackson; 2028781 and 2028782 of Jan. 28, 1936 to J. J. Jackson; **Can. Pats.** 324055 of Jul. 12, 1932 to R. G. Jackson; 325662 and 325663 of Sep. 6, 1932 to R. G. Jackson; 331193 of Mar. 21, 1933 to R. G. Jackson; 340865 of Apr. 17, 1934 to R. G. Jackson; 352870 of Sep. 10, 1935 to R. G. Jackson; **Brit. Pats.** 448498 and 448552 of Jun. 8, 1936 to R. G. Jackson; **Ger. Pat.** 529382 of Apr. 3, 1929 to Fritz Peichl.

710 [p. 695] **U. S. Pats.** 1911357 of May 30, 1933 to Dozier Finley; 1990173 of Feb. 5, 1935 to Dozier Finley.

711 [p. 695] **U. S. Pats.** 1249734 of Dec. 11, 1917 to F. B. Foster; 1273536 of Jul. 23, 1918 to George Prifold; **Brit. Pat.** 118504 of Oct. 23, 1917 to J. Y. Johnson.

712 [p. 695] **U. S. Pats.** 1562382, 1562383, 1562384, 1562385, 1562386 and 1562387 of Nov. 17, 1925 to C. M. Taylor; 1688239 of Oct. 16, 1928 to C. O. Karpf; 1765588 of Jun. 24, 1930 to R. G. Jackson; 1793666 of Feb. 24, 1931 to J. T. Baldwin; 1807291 of May 26, 1931 to R. G. Jackson; 1848329 of Mar. 8, 1932 to Carleton Ellis; 1905827 of Apr. 25, 1933 to Carleton Ellis; **Can. Pats.** 256979, 256980, 256981, 256982 and 256983 of Jan. 5, 1926 to C. M. Taylor; 296157 of Dec. 31, 1928 to Bird & Son, Inc.; 322191 of May 10, 1932 to R. G. Jackson.

713 [p. 695] **U. S. Pats.** 1784135 of Dec. 9, 1930 to Carleton Ellis; 1784799, 1784800, 1784801, 1784802, 1784803 and 1784804 of Dec. 9, 1930 to Carleton Ellis; 1784805 of Dec. 9, 1930 to B. N. Lougovoy; 1856369, 1856370 and 1856371 of May 3, 1932 to J. T. Baldwin; 1959363 of May 22, 1934 to H. H. Hopkins; 1960220 of May 22, 1934 to H. H. Hopkins.

714 [p. 696] **U. S. Pats.** 1129571 of Feb. 23, 1915 to C. F. Humphreys; 1132647 of Mar. 23, 1915 to E. L. Blabon; 1292705 of Jan. 28, 1919 to J. T. Cheney; 1899416 of Feb. 28, 1933 to D. L. Irwin and R. E. Tippins; 1940306 of Dec. 19, 1933 to W. F. Kaufman and J. C. McCarthy; **Can. Pat.** 314792 of Sep. 1, 1931 to Armstrong Cork Co.; **Brit. Pats.** of 1885 (May 23), 6314 to T. J. Flynn; 248058 of Nov. 25, 1924 to J. C. Leech; 299178 of Oct. 13, 1927 to D. L. Irwin and R. E. Tippins; 308504 of May 1, 1928 to M. H. Tate; 317375 of Aug. 14, 1929 to E. I. du Pont de Nemours & Co.; 345386 and 345387 of Jan. 14, 1930 to D. L. Irwin and R. E. Tippins; 347527 of Jul. 15, 1929 to Armstrong Cork Co.

715 [p. 696] **U. S. Pats.** 1184584 of May 23, 1916 to W. D. Snow; 1664589 of Apr. 3, 1928 to N. C. Campbell; 1843357 of Feb. 2, 1932 to N. L. Campbell; **Brit. Pat.** 290488 of Sep. 7, 1927 to W. F. Marvin.

716 [p. 696] **U. S. Pat.** 1774204 of Aug. 26, 1930 to H. L. Levin.

717 [p. 696] **U. S. Pat.** 1755978 of Apr. 22, 1930 to J. B. Campbell.

718 [p. 696] **U. S. Pats.** 1766879 of Jun. 24, 1930 to J. B. Campbell; Reissue 18582 of Aug. 30, 1932 to J. B. Campbell.

719 [p. 696] **U. S. Pat.** 1131595 of Mar. 9, 1915 to C. S. Bird and G. R. Wyman.

720 [p. 696] **U. S. Pat.** 1754253 of Apr. 15, 1930 to H. C. Avery; **Can. Pat.** 271210 of May 31, 1927 to H. C. Avery.

721 [p. 696] **U. S. Pat.** 2009966 of Jul. 30, 1935 to P. R. Allen; **Can. Pat.** 353447 of Oct. 8, 1935 to Building Products, Ltd.

722 [p. 696] **U. S. Pat.** 1358371 of Nov. 9, 1920 to A. C. Holzapfel; See also reference 139.

723 [p. 696] **Brit. Pat.** 367782 of Nov. 18, 1930 to I. G. Farbenindustrie, A.-G.

724 [p. 696] **U. S. Pat.** 1925302 of Sep. 5, 1933 to L. S. Case.

725 [p. 696] **U. S. Pat.** 1971036 of Aug. 21, 1934 to J. E. Graham and E. M. Lines; **Can. Pat.** 349111 of Mar. 26, 1935 to J. E. Graham and E. M. Lines.

726 [p. 696] **U. S. Pats.** 1360663 of Nov. 30, 1920 to J. S. Miller, Jr.; 1729832, 1729833, 1729834 and 1729835 of Oct. 1, 1929 to R. G. Jackson; 1793666 of Feb. 24, 1931 to J. T. Baldwin.

727 [p. 696] **U. S. Pats.** 1184584 of May 23, 1916 to W. D. Snow; 1378498 of May 17, 1921 to J. J. Tomec; 1427655 of Aug. 29, 1922 to W. G. Thomas, Jr.; 1664589 of Apr. 3, 1928 to N. L. Campbell; 1755978 of Apr. 22, 1930 to J. B. Campbell; 1765589 of Jun. 24, 1930 to R. G. Jackson; 1766811 of Jun. 24, 1930 to N. L. Campbell; 1887979 of Nov. 15, 1932 to H. L. Levin.

728 [p. 696] **U. S. Pats.** 1537950 of May 19, 1925 to Lester Kirschbraun; 1725646 of Aug. 20, 1929 to Lester Kirschbraun.

729 [p. 696] **U. S. Pats.** 1552852 of Sep. 8, 1925 to H. C. Innes; 1578663 of Mar. 30, 1926 to H. C. Innes.

730 [p. 696] **U. S. Pats.** 1409768 of Mar. 14, 1922 to R. P. Perry; 1611330 of Dec. 21, 1926 to C. S. Bird; **Can. Pat.** 247636 of Mar. 17, 1925 to C. S. Bird.

731 [p. 696] **U. S. Pats.** 1474423 of Nov. 20, 1923 to J. S. Maynard; 1655872 of Jan. 10, 1928 to T. J. Mell.

732 [p. 696] **U. S. Pat.** 1908414 of May 9, 1933 to Dozier Finley.

733 [p. 696] **U. S. Pats.** 1610019 of Dec. 7, 1926 to W. H. Lowe; 1929279 of Oct. 3, 1933 to A. H. Irving and O. H. Robertson.

734 [p. 696] **U. S. Pats.** 1255095 of Jan. 29, 1918 to R. G. Jackson; 1552852 of Sep. 8, 1925 to H. C. Innes; 1558383 of Oct. 20, 1925 to W. M. Mathes; **Brit. Pat.** 298862 of Mar. 29, 1928 to C. A. E. Leech.

735 [p. 697] **U. S. Pat.** 819450 of May 1, 1906 to F. C. Overbury.

736 [p. 697] "Spec. for Drill (Unbleached)," Fed. Spec. Board Spec. 557 (Bureau of Standards Circular C-267); **U. S. Pats.** 1347095 of Jul. 20, 1920 to A. B. Harrison; 1942971 of Jan. 9, 1934 to M. L. Morse.

737 [p. 697] **U. S. Pat.** 1908414 of May 9, 1933 to Dozier Finley; **Brit. Pat.** 419973 of Aug. 24, 1933 to Vereinigte Deutsche Metallwerke A.-G.

738 [p. 697] **U. S. Pat.** 1899385 of Feb. 28, 1933 to Dozier Finley.

739 [p. 697] **U. S. Pats.** 1609937 and 1609938 of Dec. 7, 1926 to C. N. Forrest; **Can. Pats.** 278535 and 278536 of Mar. 13, 1928 to Barber Asphalt Co.; **Ger. Pats,** 362727 of Jun. 9, 1920 to Barber Asphalt Paving Co.; 558849 of Aug. 27, 1926 to C. N. Forrest.

740 [p. 698] **U. S. Pat.** 1846185 of Feb. 23, 1932 to Max Cline and R. A. Masten; **Brit. Pats.** of 1838 (Jul. 11), 7731 to John Bethell; of 1862 (Nov. 27), 3184 to W. Clark; **Ger. Pat.** 221931 of May 15, 1908 to Thorvald Möller & Co.; **Danish Pat.** 10936 of Jun. 3, 1908 to Thorvald Möller & Co.

741 [p. 698] **U. S. Pats.** 1026855 of May 21, 1912 to Carleton Ellis; 1947451 and 1947452 of Feb. 20, 1934 to W. R. Barber, A. G. Natwick and M. W. Phelps; Reissue 19643 of Jul. 16, 1935 to W. R. Barber, A. G. Natwick and M. W. Phelps; 2047975 of Jul. 21, 1936 to Leo Liberthson; **Can. Pats,** 340974 of Apr. 17, 1934 to L. Sonneborn Sons, Inc.; 345855 of Nov. 6, 1934 to Pacific Mills, Ltd.; 346222 of Nov. 20, 1934 to Pacific Mills, Ltd.

742 [p. 698] **U. S. Pats,** 2067046 and 2067047 of Jan. 5, 1937 to G. H. Ellis; **Brit. Pat.** of 1893 (Sep. 18), 17514 to W. Smith.

743 [p. 698] **Brit. Pat.** of 1854 (Sep. 12), 1983 to Edward Gillman.

744 [p. 698] **Brit. Pat.** of 1806 (Mar. 8), 2913 to Patrick Whytlock.

745 [p. 698] **Swiss Pat.** 41567 of May 18, 1908 to Thorvald Möller & Co.

746 [p. 698] **U. S. Pat.** 1884367 of Oct. 25, 1932 to D. M. Sutherland, Jr.

747 [p. 698] **U. S. Pat.** 1935196 of Nov. 14, 1933 to E. C. Lathrop and F. A. Irvine.

748 [p. 698] **U. S. Pat.** 1942706 of Jan. 9, 1934 to F. A. Irvine.

749 [p. 698] "A New Wood Preservative of General Utility," by R. Falck and S. Kamesam, *Chem.-Ztg.*, **55**, 837 (1931).

750 [p. 698] **U. S. Pat.** 2022231 of Nov. 26, 1935 to F. E. Cislak.

751 [p. 698] **Ger. Pat.** Appl. M-104449 of Apr. 21, 1928 to F. A. Malchow A.-G. (rejected).

752 [p. 698] "Coal Tar and Asphalt Products for Waterproofing," by S. T. Wagner, *Chem. Eng.*, **18**, 224 (1914).

753 [p. 698] "Tentative Specifications for Creosote Oil for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level" (D 43-36T), A.S.T.M. Tentative Standards 1936, 846. **Ger. Pat.** Appl. W-83153 of Jul. 10, 1929 to Westdeutsche Asphaltwerke Vereinigte Strassenbaugesellschaften J. S. Kahlbetzer & Rheinische Asphaltgesellschaft Adam Meyer & Co., A.-G.

754 [p. 699] "Tentative Specs. for Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level" (D 41-36T), A.S.T.M. Tentative Standards 1936, 848; "Federal Spec. for Asphalt-Primer; (for) Roofing and Waterproofing," Federa

Standard Stock Catalog, Section IV, (Part 5), SS-A-701, Aug. 1, 1933, Bureau of Standards Circular C-162.

755 [p. 699] **U. S. Pat.** 819450 of May 1, 1906 to F. C. Overbury.

756 [p. 700] **Brit. Pat.** 426970 of Apr. 12, 1935 to Durastic Bituminous Products, Ltd.

757 [p. 701] "The Waterproofing of Solid Steel-floor Railroad Bridges," by S. T. Wagner, *Proc. Am. Soc. Civil Eng.*, **79**, 306 (1915); **Brit. Pat.** of 1860 (Jul. 25), 1803 to J. Pilkington.

758 [p. 701] **U. S. Pats.** 1973522 of Sep. 11, 1934 to S. W. Chaffee and R. P. Muller; 1973523 of Sep. 11, 1934 to R. P. Muller; 2043188 of Jun. 2, 1936 to Max Walten; **Can. Pats.** 361078 and 361079 of Oct. 13, 1936 to Barrett Co.; **Brit. Pat.** 455408 of Dec. 16, 1935 to G. Colmant; **Ger. Pat.** 629422 of Aug. 14, 1932 to Richard Kellerhoff, Jr.

759 [p. 702] **U. S. Pat.** 1207726 of Dec. 12, 1916 to C. V. Eades.

760 [p. 702] "Modern Methods of Waterproofing," by M. H. Lewis, Norman W. Henley Publishing Co., New York (1914); "Waterproofing Engineering," by Joseph Ross, John Wiley & Sons, Inc., New York (1919).

761 [p. 702] "Asphaltpapiere nach neuren Herstellungsverfahren," by Fritz Hoyer, *Kunstoffe*, **22**, 11 (1922); "Teerpapiere," by A. Chlorus, *Wochenblatt für Papierfabrikation*, **60**, 102 (1929); "Asphaltische Isolierpapiere, ihre Herstellung und Verwendung," by Anon., *Teer u. Bitumen*, **28**, 205 (1930); "Asphaltpapiere nach neureen Herstellungsverfahren," by Fritz Hoyer, *Asphalt u. Teer*, **31**, 41 (1931); "Bitumenpapiere und Bitumenpappen," by F. Huth, *Kartonnagen u. Papierwarenzeitung*, **37**, 738 and 754 (1933); "Die Asphaltpapiere, ihre Herstellung, Eigenschaften und Anwendungsmöglichkeiten," by Fritz Hoyer, *Bitumen*, **5**, 191 and 216 (1935); "The Improvement of Paper and Paper Board with Asphalt Emulsion," by Fritz Hoyer, Gelatine, Leim & Klebstoffe, **3**, 208 (1935); "Verwendungsmöglichkeiten, Eigenschaften und Herstellung der im Heissverfahren, erzeugten Bitumenpapiere," by E. B. Hoffmann, *Bitumen*, **6**, 34 (1936).

762 [p. 703] "Portland Cement Sidewalk Construction," by Universal Portland Cement Co., Chicago-Pittsburg, pp. 45–46 (1908); "Concrete Floors and Sidewalks," by A. A. Houghton, Norman W. Henley Publishing Co., N. Y., pp. 36–38 (1914); "A Study of Methods of Curing Concrete Pavements," by F. H. Jackson and E. W. Bauman, Public Roads, **11** (11), U. S. Dept. Agri., Bureau of Public Roads, Wash., D. C. (Jan. 1931); **U. S. Pats.** 1672380 of Jun. 5, 1928 to R. B. Gage; 1694588 of Dec. 11, 1928 to Dozier Finley and W. L. Rhodes; Reissue 17737 of Jul. 15, 1930 to R. B. Gage; 1841928 of Jan. 19, 1932 to E. T. Anderson; 1841931 of Jan. 19, 1932 to L. N. Weber; 1965111 of Jul. 3, 1934 to E. H. Angier; 1992110 of Feb. 19, 1935 to Lester Wittenberg; 1999152 of Apr. 23, 1935 to Dozier Finley; 2003988 of Jun. 4, 1935 to E. H. Angier; 2024721 of Dec. 17, 1935 to Dozier Finley and W. R. Greig; **Can. Pats.** 313550 of Jul. 21, 1931 to Texas Co.; 319009 of Jan. 19, 1932 to Cecil Morris; 322636 and 322637 of May 24, 1932 to American Reenforced Paper Co.; 326000 of Sep. 13, 1932 to International Bitumen Emulsions Corp.; **Brit. Pat.** 375196 of Nov. 25, 1931 to A. H. Stevens.

763 [p. 703] **U. S. Pat.** 1934651 of Nov. 7, 1933 to E. T. Anderson.

764 [p. 703] **Can. Pat.** 151023 of Oct. 14, 1913 to J. H. White.

765 [p. 704] "Federal Spec. for Paper (General Specs.)," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-31, Mar. 1, 1932; "Federal Spec. for Paper; Manila, Rope," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-346, "Federal Spec. for Paper; Manila, Sulphite," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-351, "Federal Spec. for Paper; Manila, Wood," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-356, "Federal Spec. for Paper; Wrapping, Kraft, No. 1," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-601, "Federal Spec. for Paper; Wrapping, Kraft, No. 2," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-606.

766 [p. 705] The addition of rag fibers rarely exceeds 25 per cent by weight.

767 [p. 706] "Federal Spec. for Paper; Kraft," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-268, May 2, 1933.

768 [p. 706] "Waterproofing of Paper," by Hermann Wandrowsky, *Papier Ztg.*, **40**, 500–1, 524–5, 579–80, 597–8, 615–6, 653–4 (1915); "A Study of Sheathing Papers," by F. T.

Carson and F. V. Worthington, Research Paper 85, Bureau of Standards, Washington, D. C. (July 1929); "Impregnation of Paper and Associated Processes," by E. E. Halls, *Ind. Chem.*, 12, 526 (1936).

769 [p. 706] **U. S. Pats.** 378520 of Feb. 28, 1888 to T. J. Pearce and M. W. Beardsley; 1492408 of Apr. 29, 1924 to P. E. Ullrich; 2071360 of Feb. 23, 1937 to James Reid; **Brit. Pat.** of 1888 (Mar. 12), 3787 to T. J. Pearce and M. W. Beardsley.

770 [p. 706] **U. S. Pat.** 1667691 of Apr. 24, 1928 to James Reid.

771 [p. 706] "Federal Spec. for Paper; Paraffin, White," Federal Standard Stock Catalogue, Section IV, (Part 5), UU-P-411.

772 [p. 706] "Federal Spec. for Paper; Kraft, Wrapping, Paraffined," UU-P-270, Federal Standard Stock Catalog, Section IV (Part 5), Sep. 30, 1936; **U. S. Pats.** 823246 of Jun. 12, 1906 to Julius Wessel; 1344620 of Jun. 29, 1920 to F. B. Davidson; 1499291 of Jun. 24, 1924 to H. M. Castner; **Can. Pats.** 30021 of Oct. 20, 1888 to W. H. Fay; 326849 of Oct. 18, 1932 to J. M. Driver; **Brit. Pats.** of 1862 (Jan. 8), 55 to John Stenhouse; of 1884 (Oct. 21), 13898 to Josiah Jowitt; of 1887 (Nov. 19), 15896 to T. F. Wiley; of 1896 (Aug. 28), 19006 to C. H. Arnold.

773 [p. 706] **U. S. Pats.** 328957 and 328958 of Oct. 27, 1885 to C. A. Maxfield.

774 [p. 706] **U. S. Pats.** 347200 of Aug. 10, 1886 to F. C. Robinson and W. H. Cothren; 369700 of Sep. 13, 1887 to W. H. Fay; 1401524 of Dec. 27, 1921 to Joaquin Crespo; 1405220 of Jan. 31, 1922 to J. R. Hyer; 1882212 of Oct. 11, 1932 to J. A. De Cew.

775 [p. 706] **U. S. Pat.** 1204149 of Nov. 7, 1916 to O. E. Gelertsen.

776 [p. 706] **U. S. Pat.** 426633 of Apr. 29, 1890 to H. J. Bird.

777 [p. 706] **U. S. Pat.** 1326079 of Dec. 23, 1919 to M. B. Mayer.

778 [p. 706] **U. S. Pat.** 1399724 of Dec. 6, 1921 to O. H. Brauser and M. H. Oakes; **Brit. Pat.** of 1865 (Jul. 29), 1962 to F. A. Abel.

779 [p. 707] **Ger. Pat.** 66892 of Jan. 17, 1893 to John Robson.

780 [p. 707] **U. S. Pat.** 1386711 of Aug. 9, 1921 to W. V. Lander.

781 [p. 707] **U. S. Pat.** 1336528 of Apr. 13, 1920 to J. W. Low.

782 [p. 707] **U. S. Pat.** 1752933 of Apr. 1, 1930 to F. W. Sullivan, Jr.

783 [p. 707] **U. S. Pats.** 1228580 of Jun. 5, 1917 to G. W. Miles; 1266955 and 1266956 of May 21, 1918 to Bruno Kniffler; 1344620 of Jun. 29, 1920 to F. B. Davidson; 1468959 of Sep. 25, 1923 to A. L. Clapp; 1499291 of Jun. 24, 1924 to H. M. Castner; 1592294 of Jul. 13, 1926 to A. L. Clapp; 1607517, 1607518 and 1607519 of Nov. 16, 1926 to Carleton Ellis; 1607552 of Nov. 16, 1926 to A. F. Maze; 1668760 of May 8, 1928 to A. L. Clapp; 1753690 of Apr. 8, 1930 to G. A. Brown; 1762928, 1762929, 1762930 and 1762931 of Jun. 10, 1930 to G. J. Manson; 1769513 of Jul. 1, 1930 to A. M. Hinkson; 1793839 of Feb. 24, 1931 to A. L. Clapp; 1802561 of Apr. 28, 1931 to Lester Kirschbraun; 1848658 and 1848659 of Mar. 8, 1932 to H. R. Rafton; 1894567 of Jan. 17, 1933 to G. J. Manson; 1899086 of Feb. 28, 1933 to Carleton Ellis; 1897694 of Feb. 14, 1933 to Carleton Ellis; 1903787 of Apr. 18, 1933 to W. C. Lodge; 1939616 of Dec. 12, 1933 to H. L. Becher; 1942438 of Jan. 9, 1934 to G. J. Manson; 1953085 of Apr. 3, 1934 to G. J. Manson; 1983918 of Dec. 11, 1934 to O. F. Neitzke; 2009488 of Jul. 7, 1935 to A. H. French; **Can. Pats.** 250680 of Dec. 8, 1925 to Lester Kirschbraun; 327574 of Nov. 15, 1932 to G. J. Manson; 342413 of Jun. 19, 1934 to Bennett, Inc.; **Brit. Pats.** 312909 of May 17, 1929 to N. V. de Bataafsche Petroleum Maatschappij and H. D. Elkington; 319745 of Sep. 27, 1928 to A. L. Clapp; 363372 of Jun. 6, 1930 to G. J. Manson; **Ger. Pats.** 201228 of Feb. 11, 1906 to H. F. M. Zanders; 303341 of Sep. 20, 1916 to J. W. Zanders; 304205 of Aug. 2, 1917 to P. E. Altmann; 305678 of Jan. 12, 1917 to P. Klemm; 307111 of Dec. 16, 1916 to Farbenfabriken vorm. Fr. Bayer & Co.; 310076 of May 8, 1917 to P. Klemm; 350622 of Oct. 23, 1917 to H. T. Böhme, A.-G., Chem. Fabrik; 352506 of Apr. 17, 1918 to H. T. Böhme, A.-G., Chem. Fabrik; 359406 of Mar. 25, 1917 to H. T. Böhme, A.-G., Chem. Fabrik; 364000 of May 4, 1918 to H. T. Böhme, A.-G., Chem. Fabrik; 376761 of Aug. 27, 1918 to Fr. Kelting; 409690 of Dec. 31, 1921 to Deutscher Erdöl, A.-G.; Appl. Sch-95268 of Aug. 31, 1931 to F. Schacht, G.m.b.H.; **Austrian Pat.** 138391 of Jul. 25, 1934 to B. R. Ruiss.

784 [p. 707] **U. S. Pat.** 1327239 of Jan. 6, 1920 to P. W. Kiernan.

785 [p. 707] **U. S. Pats.** 1800551 of Apr. 14, 1931 to G. J. Manson; 1848658 and 1848659

of Mar. 8, 1932 to H. R. Rafton; 1884038 of Oct. 25, 1932 to G. J. Manson; 1894566 of Jan. 17, 1933 to G. J. Manson; 1894959 of Jan. 24, 1933 to G. J. Manson; 2030385 of Feb. 11, 1936 to G. J. Manson; **Ger. Pat.** 332802 of Jun. 14, 1918 to W. Schwarz.

786 [p. 707] **Brit. Pat.** 359946 of May 27, 1930 to B. Redlich and Elektrochemische Werke München, A.-G.

787 [p. 707] **Can. Pats.** 315198 of Sep. 15, 1931 to Agasote Millboard Co.; 316486 of Oct. 27, 1931 to Agasote Millboard Co.

788 [p. 707] **U. S. Pat.** 691952 of Jan. 28, 1902 to A. D. Little.

789 [p. 707] **U. S. Pat.** 1842112 of Jan. 19, 1922 to H. P. Pearson.

790 [p. 707] **U. S. Pats.** 1948442 of Feb. 20, 1934 to Carleton Ellis; 2042299 of May 26, 1936 to Carleton Ellis; 2043992 of Jun. 16, 1936 to Carleton Ellis.

791 [p. 707] **U. S. Pat.** 1880971 of Oct. 4, 1932 to T. B. Munroe and E. C. Lathrop.

792 [p. 707] **U. S. Pat.** 1981608 of Nov. 20, 1934 to Harry Bennett.

793 [p. 707] **U. S. Pat.** 1860466 of May 31, 1932 to Gustave Landt and H. L. Becher; **French Pat.** 739751 of Jul. 7, 1932 to Bitumen Investments, Inc.

794 [p. 707] **U. S. Pat.** 1884110 of Oct. 25, 1932 to F. G. Morehouse.

795 [p. 707] **U. S. Pat.** 517732 of Apr. 3, 1894 to W. H. H. Childs.

796 [p. 707] **U. S. Pat.** 426633 of Apr. 29, 1880 to H. J. Bird.

797 [p. 707] **U. S. Pat.** 378520 of Feb. 28, 1888 to T. J. Pearce and M. W. Beardsley.

798 [p. 707] **U. S. Pats.** 2036301 of Apr. 7, 1936 to H. J. Rose; 2054112, 2054113 and 2054114 of Sep. 15, 1936 to Allen Abrams and C. L. Wagner; 2054115 and 2054116 of Sep. 15, 1936 to Allen Abrams, C. L. Wagner and G. W. Forcey; **Can. Pat.** 363562 of Jan. 19, 1937 to Marathon Paper Mills Co.; 363890 of Feb. 2, 1937 to Marathon Paper Mills Co.

799 [p. 707] **U. S. Pat.** 1715080 of May 28, 1929 to O. A. Frederickson.

800 [p. 708] **U. S. Pats.** 52788 of Feb. 20, 1866 to Stuart Gwynn; 66302 of Jul. 2, 1867 to L. M. Crane; 89094 and 89095 of Apr. 20, 1869 to Charles Toppan; 89738 of May 4, 1869 to Charles Cowan; 123747 of Feb. 13, 1872 to J. H. Tiemann; 147833 of Feb. 24, 1874 to A. G. Fell; 149319 of Apr. 7, 1874 to Xavier Karcheski; 193867 of Jun. 4, 1877 to Siegfried Hammerschlag; 196531 of Oct. 30, 1877 to Oliver Long; 209393 of Oct. 29, 1878 to Siegfried Hammerschlag; 217280 of Jul. 8, 1879 to Siegfried Hammerschlag; 233282 of Oct. 12, 1880 to G. H. Rich; 243353 of Jun. 28, 1881 to J. T. Bedford; 247841 of Sep. 27, 1881 to O. R. Chase; 254105 of Feb. 28, 1882 to J. T. Bedford; 257761 of May 9, 1882 to J. H. Ridgway; 278443 of May 29, 1883 to C. A. Maxfield; 286403 of Oct. 9, 1883 to J. B. Duke; 303309 of Sep. 2, 1884 to Herman Frasch; 318911 of May 26, 1885 to Josiah Jowitt; Reissue 10618 of Jun. 30, 1885 to C. A. Maxfield; 326687, 326688 and 326689 of Sep. 22, 1885 to E. G. Sparks; 328957 and 328958 of Oct. 27, 1885 to C. A. Maxfield; 329743 of Nov. 3, 1885 to W. B. Howe; 340810 of Apr. 27, 1886 to F. W. Miller and J. J. Newman; 341587 of May 11, 1886 to William Macrone; 343345 of Jun. 8, 1886 to E. G. Sparks; 343375 of Jun. 8, 1886 to W. B. Howe; 344142 of Jun. 22, 1886 to E. G. Sparks; 344965 of Jul. 6, 1886 to W. B. Howe and M. D. Knowlton; 345236 of Jul. 6, 1886 to John Jordan; 346869 of Aug. 3, 1886 to Isaac Sherck and Joseph Batig, Jr.; 349462 of Sep. 21, 1886 to E. G. Sparks; 349530 and 349531 of Sep. 21, 1886 to Isaac Sherck and Joseph Batig, Jr.; 351748 of Nov. 2, 1886 to A. H. Dingman; 353122 of Nov. 23, 1886 to S. M. Chester; 360373 of Mar. 29, 1887 to S. M. Chester; 362547 of May 10, 1887 to Isaac Sherck and Joseph Batig, Jr.; 365213 of Jun. 21, 1887 to C. A. Wilkinson; 367886 of Aug. 9, 1887 to D. A. Andrews; 374025 of Nov. 15, 1887 to C. A. Wilkinson and W. S. McDonald; 394268 of Dec. 11, 1888 to C. E. Haynes; 395645 of Jan. 1, 1889 to J. B. Anderson; 399544 of Mar. 12, 1889 to Thomas Manahan; 405044 of Jun. 11, 1889 to George Manahan and H. Gade; 409892 of Aug. 27, 1889 to O. Woodworth and H. O. Woodworth; 413286 of Oct. 22, 1889 to F. W. Miller and J. J. Newman; 437435 of Sep. 30, 1890 to W. B. Howe; 453090 of May 26, 1891 to J. J. Newman; 497198 of May 9, 1893 to J. J. Newman; 513030 of Jan. 11, 1894 to J. W. Bliss and J. C. Erwin; 528752 of Nov. 6, 1894 to A. S. Woodward; 532127 of Jan. 8, 1895 to C. C. Macbrair; 559605 of May 5, 1896 to L. G. Reynolds; 606869 of Jul. 5, 1898 to C. C. Macbrair; 670518 of Mar. 26, 1901 to C. F. W. Schmacher; 677320 of Jun. 25, 1901 to S. R. Norris and E. H. Vavra; 690868 of Jan. 7, 1902 to James Menzies; 721693 of Mar. 3, 1903 to C. E. Goessmann; 728234 of May 19, 1903 to C.

C. Hoyt; 737826 of Sep. 1, 1903 to R. J. Carrier; 785110 of Mar. 21, 1905 to T. E. Kinney; 826518 of Jul. 17, 1906 to J. B. Morrow; 836336 of Nov. 20, 1906 to J. O. Parker; 836743 of Nov. 27, 1906 to Richard Falter; 858149 of Jun. 25, 1907 to Martin Cantine; 873126 of Dec. 10, 1907 to H. P. Hilbert; 875241 of Dec. 31, 1907 to Martin Cantine; 883359 of Mar. 31, 1908 to C. F. Spaulding; 890221 of Jun. 9, 1908 to F. B. Davidson and William Gorges; 894366 of Jul. 28, 1908 to Martin Cantine; 915672 of Mar. 16, 1909 to A. W. Harrington; 920529 of May 4, 1909 to A. J. Bradley; 940969 of Nov. 23, 1909 to P. Hinkle; 951293 of Mar. 8, 1910 to J. C. Thompson; 953570 of Mar. 29, 1910 to W. A. Zickerman; 1042914 of Oct. 29, 1912 to H. R. Hoyl; 1043021 of Oct. 29, 1912 to C. W. Mayer; 1063441 of Jun. 3, 1913 to A. W. Harrington; 1140873 of May 25, 1915 to W. L. Carter; 1152136 of Aug. 31, 1915 to G. R. Wyman; 1167403 of Jan. 11, 1916 to F. H. Noberg; 1185600 of May 30, 1916 to F. Holt; 1197925 of Sep. 12, 1916 to S. Elliott; 1198976 of Sep. 19, 1916 to E. H. Vavra; 1203302 and 1203303 of Oct. 31, 1916 to C. A. Bickett; 1276439 of Aug. 20, 1918 to L. W. Sutherland; 1277904 of Sep. 3, 1918 to H. Gesell; 1283851 of Nov. 5, 1918 to C. W. Mayer; 1288641 and 1288642 of Dec. 24, 1918 to C. W. Mayer; 1299388 of Apr. 1, 1919 to W. H. Scott; 1321925 of Nov. 18, 1919 to E. L. Lake; 1336528 of Apr. 13, 1920 to J. W. Low; 1345720 of Jul. 6, 1920 to F. P. Vavra; 1347619 of Jul. 27, 1920 to F. B. Davidson; 1367274 of Feb. 1, 1921 to G. W. Miles; 1391281 of Sep. 20, 1921 to G. C. Snyder; 1791040 of Feb. 3, 1931 to W. H. Richardson; 1880065 of Sep. 27, 1932 to E.P. Arpin, Jr.; 1880078 of Sep. 27, 1932 to W. J. Eisner; 1963058 of Jun. 12, 1934 to V. H. Wilshire; **Can. Pat.** 125900 of May 24, 1910 to A. W. Harrington.

801 [p. 708] **U. S. Pats.** 91133 of Jun. 8, 1869 to James Howard; 1336528 of Apr. 13, 1920 to J. W. Low.

802 [p. 708] **U. S. Pats.** 1595637 of Aug. 10, 1926 to M. W. Wheildon; 1667691 of Apr. 24, 1928 to James Reid; **Ger. Pat.** 514922 of Nov. 24, 1927 to Scutan Co.

803 [p. 708] **U. S. Pat.** 1268446 of Jun. 4, 1918 to G. E. Ferguson; **Brit. Pat.** 340749 of Dec. 3, 1929 to E. Rudin and Heinrich Kollbrunner.

804 [p. 709] **U. S. Pat.** 1944907 of Jan. 30, 1934 to F. H. McCormick and G. L. Schwartz.

805 [p. 709] **U. S. Pat.** 1772436 of Aug. 5, 1930 to O. A. Frederickson.

806 [p. 709] **Brit. Pat.** 318412 of Oct. 15, 1928 to I. G. Farbenindustrie, A.-G.

807 [p. 709] **U. S. Pat.** 1358834 of Nov. 16, 1920 to F. W. Farrell.

808 [p. 709] **U. S. Pats.** 361050 of Apr. 12, 1887 to W. H. H. Childs; 372894 of Nov. 8, 1887 to W. H. H. Childs; 708454 of Sep. 2, 1902 to C. S. Bird and J. B. Hanscom; 775968 of Nov. 29, 1904 to August Gross; 784831 of Mar. 14, 1905 to C. S. Bird; 1154875 of Sep. 28, 1915 to James Meade; 1320549 of Nov. 4, 1919 to O. A. Heppes; 1341955 of Jun. 1, 1920 to J. J. White; 1353323 of Sep. 21, 1920 to F. B. Davidson; 1503613 of Aug. 5, 1924 to W. L. Taylor; 1789887 of Jan. 20, 1931 to W. L. Taylor; 1882715 of Oct. 18, 1932 to E. H. Angier; 1895527 of Jan. 31, 1933 to J. H. Swan; 2031035 of Feb. 18, 1936 to C. G. Dreymann; **Can. Pats.** 323002 of Jun. 7, 1932 to J. A. Lowe; 335334 of Sep. 5, 1933 to P. B. Burchardt; **Brit. Pats.** of 1845 (Jul. 21), 10774 to T. R. Williams; 387310 of Apr. 27, 1931 to Flintkote Co.; 393790 of Jun. 15, 1933 to P. B. Burchardt; 399304 of Oct. 5, 1933 to Leopold Rado; 412498 of Dec. 16, 1933 to H. E. Potts; **Swedish Pat.** 76082 of Dec. 13, 1932 to P. B. Burchardt; "Spec. for Asphalt, for Use in Manufacture of Fiber Containers," 3-160, issued by the U. S. Army.

809 [p. 710] **U. S. Pats.** 670393 of Mar. 19, 1901 to James Arkell; 728828 and 728829 of May 26, 1903 to James Arkell; 1282167 of Oct. 22, 1918 to E. H. Angier; 1493371 of May 6, 1924 to W. J. Moeller; 1595637 of Aug. 10, 1926 to W. M. Wheildon; Reissue 17633 of Apr. 1, 1930 to W. W. Rowe; 1774573 of Sep. 2, 1930 to A. L. Spafford; 1782767 of Nov. 25, 1930 to W. W. Rowe; 1882714 of Oct. 18, 1932 to E. H. Angier and E. R. Dearborn; 1893717 of Jan. 10, 1933 to W. W. Rowe; 1914869 of Jun. 20, 1933 to W. W. Rowe; 1928045 of Sep. 26, 1933 to E. H. Angier; 1975286 of Oct. 2, 1934 to F. M. O. Pinoff; 1984910 of Dec. 18, 1934 to E. H. Angier; 2030156 of Feb. 11, 1936 to W. W. Rowe; 2061748 of Nov. 24, 1936 to E. H. Angier; **Can. Pats.** 285007 of Nov. 20, 1928 to Paper Service Co.; 307996 of Jan. 20, 1931 to Paper Service Co.; 308198 of Jan. 27, 1931 to Wood Conversion Co.; 323955 of Jul. 5, 1932 to Paper Service Co.; 349785 of Apr. 23, 1935 to Paper Patents Co.; **Brit. Pat.** 359154 of

Sep. 22, 1930 to W. W. Rowe; **Ger. Pat.** 580921 of Feb. 12, 1930 to Franz Schmid; 632241 of Jul. 4, 1936 to Bitumen Handelgesellschaft m.b.H.

810 [p. 710] **U. S. Pats.** 1757479 of May 6, 1930 to Ernst Schmidt; 1890418 of Dec. 6, 1932 to Ernst Schmidt; 2030668 of Feb. 11, 1936 to Frederick Weyerhaeuser and J. S. Busch; 2065402 of Dec. 22, 1936 to E. F. Schweller; **Ger. Pat.** Design 1354219 of Oct. 22, 1935 to A. W. Andernach, G.m.b.H.

811 [p. 710] **U. S. Pats.** 1195949 and 1195950 of Aug. 22, 1916 to M. R. Carney; 1211848, 1211849, 1211850, 1211851 and 1211852 of Jan. 9, 1917 to C. H. Howard; 1227520 of May 22, 1917 to E. H. Angier; 1250230 of Dec. 18, 1917 to Theodore Scherf; 1255818 of Feb. 5, 1918 to P. T. Jackson, Jr.; 1270250 of Jun. 18, 1918 to A. F. Schenkelberger; 1278471 of Sep. 10, 1918 to C. H. Howard; 1278935 of Sep. 17, 1918 to P. T. Jackson, Jr.; 1285465 of Nov. 19, 1918 to C. B. Tewksbury; 1314519 of Sep. 2, 1919 to C. H. Howard; 1316241 of Sep. 26, 1919 to C. H. Howard; 1318149, 1318150 and 1318151 of Oct. 7, 1919 to C. H. Howard; 1333718 of Mar. 16, 1920 to P. T. Jackson, Jr.; 1335058 of Mar. 30, 1920 to P. T. Jackson, Jr.; 1337702 of Apr. 20, 1920 to C. H. Howard; 1341981 of Jun. 1, 1920 to C. H. Howard; 1446092 and 1446094 of Feb. 20, 1923 to P. T. Jackson, Jr.; 1460356 of Jun. 26, 1923 to C. H. Howard; 1464155 of Aug. 7, 1923 to P. T. Jackson, Jr.; 1472021 of Oct. 23, 1923 to P. T. Jackson, Jr.; 1518512 and 1518513 of Dec. 9, 1924 to C. H. Howard; 1520349 of Dec. 23, 1924 to C. H. Howard; 1551992 of Sep. 1, 1925 to C. H. Howard; 1565008 and 1565009 of Dec. 8, 1925 to C. H. Howard; 1590167 of Jun. 22, 1926 to C. H. Howard; 1605953 of Nov. 9, 1926 to C. H. Howard; 1644050 of Oct. 4, 1927 to W. H. Cady; 1652861 of Dec. 13, 1927 to C. H. Howard; 1697721 of Aug. 7, 1928 to C. H. Howard; 1841945 and 1841946 of Jan. 19, 1932 to G. E. Gibbs; 1850301 of Mar. 22, 1932 to G. E. Gibbs; 1866513 of Jul. 5, 1932 to J. W. Ballard. 1867071 of Jul. 12, 1932 to W. R. Collings and D. L. Gibb; 1870215 of Aug. 9, 1932 to H. A. Anderson; 1871602 of Aug. 16, 1932 to G. E. Gibbs; 1885280 of Nov. 1, 1932 to H. D. Moore; 1894219 of Jan. 10, 1933 to G. E. Gibbs; 1901999 of Mar. 21, 1933 to C. A. Upson; 1906654 of May 2, 1933 to W. N. Stevenson; 1942354 of Jan. 2, 1934 to W. R. Collings and D. L. Gibb; 1962682 of Jun. 12, 1934 to William Case; 1974594 of Sep. 25, 1934 to E. H. Angier; 1984814 of Dec. 18, 1934 to J. P. Sherman; 2026194 of Dec. 31, 1935 to E. C. Smith; 2061375 of Nov. 17, 1936 to J. D. Cramer; 2069063 of Jan. 26, 1937 to G. E. Gibbs; 2075166 of Mar. 30, 1937 to C. V. Brady; **Can. Pat.** 350323 of May 21, 1935 to Canada Paper Co.; 363927 of Feb. 2, 1937 to Ole Heye; **Brit. Pats.** 372039 of Mar. 19, 1931 to Conrad Arnold; 458336 of Mar. 11, 1935 to Ole Heye; **Ger. Pats.** 222959 of Mar. 10, 1909 to Alexander Wendler; 258214 of May 7, 1912 to E. H. Angier; 552713 of Mar. 5, 1931 to Berkenhoff & Drebes, A.-G.; 624493 of Nov. 19, 1929 to Richard Doorentz; Design 1313447 of Aug. 23, 1934 to Eugen Hoeltenschmidt; Design 1401031 of Feb. 11, 1937 to Paul Lechler.

812 [p. 710] **U. S. Pats.** 1526553 of Feb. 17, 1925 to Lester Kirschbraun; 1735030 of Nov. 12, 1929 to Edmund Bache.

813 [p. 710] **U. S. Pats.** 1479043 of Jan. 1, 1924 to Lester Kirschbraun; 1526552 of Feb. 17, 1925 to Lester Kirschbraun; 1723361 of Aug. 6, 1929 to H. L. Levin; 1724137 of Aug. 13, 1929 to Lester Kirschbraun and C. R. MacDonald; 1735030 of Nov. 12, 1929 to Edmund Bache; 1851176 of Mar. 29, 1932 to A. R. Harvey.

814 [p. 710] **U. S. Pat.** 1550648 of Aug. 18, 1925 to R. P. Perry; **Can. Pat.** 233378 of Aug. 7, 1923 to Barrett Co.; **Ger. Pats.** 258214 of May 7, 1912 to E. H. Angier; Design 415347 of Feb. 24, 1920 to August Prensz.

815 [p. 710] **U. S. Pats.** 1196219 of Aug. 29, 1916 to C. F. Eckart; 1227898 of May 29, 1917 to C. F. Eckart; 1249355 of Dec. 11, 1917 to C. F. Eckart; 1274527 of Aug. 6, 1918 to C. F. Eckart; 1276887 of Aug. 27, 1918 to C. F. Eckart; 1287267 of Dec. 10, 1918 to C. F. Eckart; 1296173 of Mar. 4, 1919 to C. F. Eckart; 1372995, 1372996, 1372997 qnd 1372999 of Mar. 29, 1921 to C. F. Eckart; 1377566 of May 10, 1921 to C. F. Eckart; 1382069 of Jun. 21, 1921 to C. F. Eckart; 1396269 of Nov. 8, 1921 to C. F. Eckart; Reissue 15231 of Nov. 22, 1921 to C. F. Eckart; 1534371 and 1534372 of Apr. 21, 1925 to Dozier Finley; 1553035 of Sep. 8, 1925 to C. F. Eckart; 1562353 of Nov. 17, 1925 to N. B. McGuire and Andrew Guild, Sr.; 1584589 of May 11, 1926 to C. A. Adams; 1654242 of Dec. 27, 1927 to C. F. Eckart; 1784376 of Dec. 9, 1930 to D. P. Moore; 1807205 of May 26, 1931, to W. M. Floring; 1810463 of Jun.

16, 1931 to J. H. Cook; 1844051 of Feb. 9, 1932 to C. J. Allen; 1846185 and 1846186 of Feb. 23, 1932 to Max Cline and R. A. Masten; 1846274 of Feb. 23, 1932 to R. B. Otwell; 1870110 of Aug. 2, 1932 to W. A. Hall; 1882713 of Oct. 18, 1932 to E. H. Angier; 1910289 of May 23, 1933 to G. B. Hoag; 1929903 of Oct. 10, 1933 to W. R. Schindler; 1930939 of Oct. 17, 1933 to Albert Horner; 1931248 of Oct. 17, 1933 to C. C. Bryant; **Can. Pats.** 256470 of Dec. 22, 1925 to C. F. Eckart; 315596 of Sep. 29, 1931 to H. J. Herfort; 320190 and 320191 of Mar. 1, 1932 to International Paper Co.; 336031 of Sep. 26, 1933 to W. A. Hall; **Porto Rican Pats.** Regs. 1227898, 1287267, 1377566, 1382069 and Reissue 15231 to C. F. Eckart; **Cuban Pat.** 2964 to C. F. Eckart; **Mex. Pat.** 20141 of Apr. 2, 1921 to C. F. Eckart; **Brit. Pats.** 370482 of Jan. 2, 1931 to W. A. Hall; **Ger. Pats.** 556201 of Jun. 30, 1931 to Josef Madl; 557638 of Jun. 24, 1931 to Natronzellstoff- und Papierfabriken, A.-G.; Design 1203272 of Jan. 2, 1932 to I. G. Farbenindustrie, A.-G.; 574897 of Feb. 4, 1932 to Natronzellstoff- und Papierfabriken, A.-G.; Design 1255930 of Aug. 1, 1932 to Otto Trautloft; Appl. N-33257 of Feb. 3, 1932 to Natronzellstoff- und Papierfabriken, A.-G.

816 [p. 711] "Crop-plant Stimulation with Paper Mulch," by L. H. Flint, Tech. Bull. 75, U. S. Dept. Agriculture, Washington, D. C., (May 1928); "Papiere im Dienste der Landwirtschaft," by E. Hasel, *Papier-Journalen*, **16**, 129 (1928); "Mulch-Paper," by W. Rivise, *Paper Trade J.*, **58**, 55 (1929); "Fruchtbares Mulchpapier," by H. R. F. Stander, *Le Papier*, **33**, 1183 (1930); "Unkrautbekämfung durch Papier," by W. Knopf, *Kartonnagen- u. Papierwarenzeitung*, **34**, 1045 (1930); "Die Bodenbedeckung," by Johannes Schanerus, Verlagsbuchhandlung C. Heinrich, Dresden (1931); "Papierindustrie und Landwirtschaft," by Ch. Groud, *Le Papeterie*, **54**, 1173 (1932).

817 [p. 711] **Can. Pats.** 321705 and 321706 of Apr. 19, 1932 to Wiremould Co.; 325552, 325553 and 325554 of Aug. 30, 1932 to National Electric Products Corp.; **Brit. Pats.** 375522 of Jun. 30, 1932 to General Cable Corp.; 380113 of Jun. 5, 1931 to General Cable Corp.

818 [p. 712] **U. S. Pat.** 1944822 of Jan. 23, 1934 to W. W. Church and W. C. Robinson.

819 [p. 712] **Can. Pat.** 280224 of May 14, 1928 to Wiremould Co.

820 [p. 712] **Brit. Pat.** 375433 of Jan. 30, 1931 to Chemieprodukte, G.m.b.H.

821 [p. 712] "Improved Weather-resistant Coatings for Overhead Line Wires," by L. L. Carter, J. W. Olson, C. F. Harding and R. N. Shreve, Research Series No. 43, Engineering Experiment Station Purdue University, Lafayette, Ind. (Nov. 1932); **U. S. Pats.** 11096 of Jun. 13, 1854 to Thomas, Earl of Dundonald; 19778 of Mar. 30, 1858 to J. B. Hyde; 122962 of Jan. 23, 1872 to C. H. Pond; 148910 of Mar. 24, 1874 to Alexander Wilkinson; 227352 of May 11, 1880 to Edgar Everhart; 239466 of Mar. 29, 1881 to E. J. De Smedt; 239776 of Apr. 5, 1881 to W. T. Henley; 267045 of Nov. 7, 1882 to R. S. Waring and J. B. Hyde; 267046 of Nov. 7, 1882 to R. S. Waring; 268034 of Nov. 28, 1882 to Murdoch Mackay; 276891 of May 1, 1883 to J. G. Sanderson; Reisse 10350 of Jul. 3, 1883 to R. S. Waring and J. B. Hyde; Reissue 10351 of Jul. 3, 1883 to R. S. Waring; 281999 of Jul. 24, 1883 to J. B. Hyde; 282914 of Aug. 7, 1883 to J. F. Martin; 283044 of Aug. 14, 1883 to D. B. Turner; 283200 of Aug. 14, 1883 to H. R. Brissett and John Howe; 284098 of Aug. 28, 1883 to R. S. Waring; Reissues 10403 and 10404 of Nov. 6, 1883 to J. B. Hyde; 290057 and 290058 of Dec. 11, 1883 to J. B. Hyde; 292770 of Jan. 29, 1884 to P. H. Vander Weyde; 307184 of Oct. 28, 1884 to A. Derrom; 319079 of Jun. 2, 1885 to J. W. Ellis; 320921 of Jun. 30, 1885 to R. S. Ferguson, W. Schumacher and W. Tubman; 321956 of Jul. 14, 1885 to J. W. Ellis; 327477 of Sep. 29, 1885 to H. C. Spalding; 335495 of Feb. 2, 1886 to J. B. Williams; 336018 of Feb. 9, 1886 to W. J. Rigney and J. Wolff; 339777 of Apr. 13, 1886 to J. Howe; 348994 of Sep. 14, 1886 to T. J. Pearce and M. W. Beardsley; 351611 of Oct. 28, 1886 to R. Alexander; 352445 of Nov. 9, 1886 to J. W. Butler; 371681 of Oct. 18, 1887 to John Grant; 393209 of Nov. 20, 1888 to A. Poitevent; 401014 of Apr. 9, 1889 to Alfonso de Figaniére; 415864 of Nov. 26, 1889 to S. H. Gilson; 441870 of Dec. 2, 1890 to E. T. Greenfield and J. Nagel; 472352 of Apr. 5, 1892 to I. Hill; 515192 of Feb. 20, 1894 to G. A. Cannot; 621807 of Mar. 28, 1899 to B. Ford; 1305222 of May 27, 1919 to E. J. Kieth; 1503337 of Jul. 29, 1924 to W. R. Seigle; 1890253 znd 1890254 of Dec. 6, 1932 to O. A. Frederickson; 2018404 of Oct. 22, 1935 to L. F. Lamplough and C. E. Plass; 2029546 of Feb. 4, 1936 to R. A. Schatzel; **Can. Pats.** 37037 of Jul. 23, 1891 to J. B.

Williams; 49942 of Sep. 10, 1895 to Joseph Hoffman; 305832 of Nov. 18, 1930 to Anaconda Wire & Cable Co.; **Brit. Pat.** 435454 of Sep. 16, 1935 to Pirelli General Cable Works, Ltd.

822 [p. 712] **U. S. Pat.** 1832929 of Nov. 24, 1931 to W. W. Evans.

823 [p. 712] **Brit. Pat.** of 1883 (Jan. 24), 399 to L. A. Groth; **Ger. Pat.** 217026 of Dec. 15, 1909 to Alfred Vogelgesang.

824 [p. 713] **U. S. Pat.** 1956721 of May 1, 1934 to J. A. Kennedy.

825 [p. 713] **U. S. Pats.** 2056130 of Sep. 29, 1936 to A. S. Watson; 2072557 of Mar. 2, 1937 to A. P. Hinsky; **Can. Pats.** 304939 of Oct. 21, 1930 to Anaconda Wire & Cable Co.; 329084 of Jan. 3, 1933 to National Electric Products Corp.

826 [p. 713] **Brit. Pat.** 345330 of Dec. 17, 1929 to N. M. Loudon.

827 [p. 713] **U. S. Pat.** 1996652 of Apr. 2, 1935 to W. C. Robinson.

828 [p. 713] **U. S. Pat.** 2008277 of Jul. 16, 1935 to C. S. Gordon.

829 [p. 713] **Brit. Pats.** 436686 of Oct. 16, 1935 to Callender's Cable and Construction Co., Ltd.; 436876 of Oct. 21, 1935 to Callender's Cable and Construction Co. Ltd.

830 [p. 713] **U. S. Pats.** 3109079 of Jun. 2, 1885 to J. W. Ellis; 1854230 of Apr. 19, 1932 to M. O. Schur; 1864674 of Jun. 28, 1932 to M. O. Schur; 1878452 of Sep. 20, 1932 to C. L. Keller; 1990248 of Feb. 5, 1935 to Howard Parker; 2062832 of Dec. 1, 1936 to F. D. Saylor; 2075700 of Mar. 30, 1937 to Edmund Burke; **Can. Pat.** 322255 of May 10, 1932, to Brown Co.

831 [p. 713] **U. S. Pats.** 522946 of Jul. 10, 1894 to Jordan Shultis and D. J. Shultis; 1180625 of Apr. 25, 1916 to B. C. Vaughn; 1242903 of Oct. 16, 1917 to E. H. Angier; 1310715 of Jul. 22, 1919 to C. A. Rose and Gustav Monrath; 1316591 of Sep. 23, 1919 to R. P. Perry; 1382740 of Jun. 28, 1912 to R. P. Perry; **Can. Pats.** 254385 of Oct. 6, 1925 to Brown Co.; 273457 of Aug. 30, 1927 to D. D. Jackson; 283833 of Oct. 9, 1928 to Robert Illemann and R. A. Whitson; **Ger. Pat.** 55585 of Jul. 29, 1890 to W. Hengstenberg.

832 [p. 713] **U. S. Pat.** 1751935 of Mar. 25, 1930 to E. W. Lovering and W. B. Van Arsdel.

833 [p. 713] **Brit. Pat.** 281316 of Nov. 24, 1927 to Scutan Company.

834 [p. 713] **U. S. Pat.** 1396060 of Nov. 8, 1921 to G. A. Richter, W. B. Van Arsdel and D. H. White.

835 [p. 713] **Can. Pat.** 322458 of May 17, 1932 to Brown Co.

836 [p. 713] **U. S. Pat.** 1137043 of Apr. 27, 1915 to F. P. Wood.

837 [p. 713] **U. S. Pat.** 284794 of Sep. 11, 1883 to S. M. Allen.

838 [p. 714] **U. S. Pats.** 1277108 of Aug. 27, 1918 to W. D. Pardoe; 1301612 of Apr. 22, 1919 to Sumner Simpson; 1354996 of Oct. 5, 1920 to W. D. Pardoe; 1450319 of Apr. 3, 1923 to Lester Kirschbraun; 1578928 of Mar. 30, 1926 to Summer Simpson; 1810714 of Jun. 16, 1931 to Lester Kirschbraun; **Can. Pats.** 260598 of May 18, 1928 to Raybestos Co.; 302357 of Jul. 22, 1930 to Raybestos Co.; 321631 of Apr. 19, 1932 to Canadian Raybestos Co., Ltd.

839 [p. 714] **Can. Pat.** 233651 of Aug. 21, 1923 to W. R. Seigle.

840 [p. 714] **U. S. Pat.** 1436362 of Nov. 21, 1922 to W. R. Seigle; 2037189 of Apr. 14, 1936 to J. W. Abernethy, S. J. Smyer and A. B. Kuhn.

841 [p. 714] **U. S. Pat.** 1498386 of Jun. 17, 1924 to Lester Kirschbraun.

842 [p. 714] **U. S. Pat.** 1275043 of Aug. 6, 1918 to Lester Kirschbraun.

843 [p. 714] **U. S. Pats.** 1270559 of Jun. 25, 1918 to Sumner Simpson; 1712002 of May 7, 1929 to J. A. Heany.

844 [p. 714] **U. S. Pat.** 1941280 of Dec. 26, 1933 to Mark Shoeld.

845 [p. 714] **U. S. Pat.** 1249019 of Dec. 4, 1917 to H. St. L. Buchner.

846 [p. 714] **U. S. Pats.** 2006232 of Jun. 25, 1935 to C. A. Upson; 2043987 of Jun. 16, 1936 to G. B. Brown; 2045384 of Jun. 23, 1936 to William Gerb; 2063964 and 2064327 of Dec. 15, 1936 to C. A. Upson; **Brit. Pats.** 449979 of Jun. 26, 1935 to F. B. Dehn; 450183 of Feb. 11, 1935 to William Gerb.

847 [p. 714] **U. S. Pats.** 2008654 and 2008655 of Jul. 16, 1935 to G. W. Clarvoe; 2028950 of Jan. 28, 1936 to M. S. Randall; 2028962 of Jan. 28, 1936 to H. J. Woodall and M. S. Randall; 1975918 of Oct. 9, 1934 to R. E. Berg; **Can. Pat.** 361149 of Oct. 13, 1936 to Johns-Manville Corp.

848 [p. 714] **U. S. Pat.** 2019233 of Oct. 29, 1935 to G. A. Nicol, Jr.; **Can. Pat.** 359961 of Aug. 18, 1936 to G. A. Nicol, Jr.

849 [p. 714] **U. S. Pats.** 1494346 of May 20, 1924 to F. H. Eastman and G. R. Stark; 2063611 of Dec. 8, 1936 to W. J. MacLeod; **Can. Pats,** 247139 of Feb. 24, 1925 to F. H. Eastman; 248327 of Mar. 31, 1925 to F. H. Eastman and G. R. Stark.

850 [p. 714] **U. S. Pat.** 2037205 of Apr. 14, 1936 to L. F. Barnum.

851 [p. 715] **Brit. Pat.** 412393 of Jun. 28, 1934 to R. E. Horley.

852 [p. 716] "Tentative Specs. and Tests for Friction Tape for General Use for Electrical Purposes" (D 69–36T), A.S.T.M. Tentative Standards 1936, 1048; "Tentative Specs. and Tests for Rubber Insulating Tape" (D 119–35T), A.S.T.M. Tentative Standards 1936, 1054; "Tentative Specs. for Tolerances and Test Methods for Woven Tapes" (D 259–36T), A.S.T.M. Tentative Standards 1936, 1206; "Standard Specifications for 0.007-in. Cotton Tape for Electrical Purposes," (D 335–36), A.S.T.M. Standards 1936, II, 1304; "Tentative Methods of Test for Thickness of Solid Electrical Insulation" (D 374–36T), A.S.T.M. Tentative Standards 1936, 1011; "Standard Specs. and Test Methods for Asbestos Tape for Electrical Purposes" (D 315–36), A.S.T.M. Standards 1936, II, 1324; "Federal Spec. for Tape; Friction," Federal Standard Stock Catalog, Section IV, (Part 5), HH-T-101, Mar. 4, 1930.

853 [p. 717] **U. S. Pat.** 1842616 of Jan. 26, 1932 to I. W. Levine.

854 [p. 717] **Can. Pat.** 340705 of Apr. 10, 1934 to W. W. McLaurin.

855 [p. 717] "Study of Commercial Wall Boards," by F. C. Clark and A. D. Conley, *Paper*, **25**, No. 23 (Feb. 11, 1920); "Properties of Fiber Building Boards," by C. G. Weber, F. T. Carson and L. W. Snyder, Misc. Publication No. 132, Bureau of Standards, Wash., D. C. (1931).

856 [p. 717] **U. S. Pats.** 1033756 of Jul. 23, 1912 to W. G. Fiske; 1292067 of Jan. 21, 1919 to H. H. Robertson; 1353323 of Sep. 21, 1920 to F. B. Davidson; 1503957 of Aug. 5, 1924 to Otto Kress; 1689812 of Oct. 30, 1928 to F. P. Wood; 1694523 of Dec. 11, 1928 to J. F. White; 1905397 of Apr. 25, 1933 to C. G. Reynolds; 1910671 of May 23, 1933 to S. J. Blum;

857 [p. 717] **U. S. Pats.** 269786 of Dec. 26, 1882 to S. H. Hamilton; 1033756 of Jul. 23, 1912 to W. G. Fiske; 1541587 of Jan. 9, 1925 to Joseph Regenstein; 1569107 of Jan. 12, 1926 to J. F. White; 1656647 of Jan. 17, 1928 to Joseph Regenstein; 1694523 of Dec. 11, 1928 to J. F. White; 2073894 of Mar. 16, 1937 to F. P. Wood.

858 [p. 717] **U. S. Pats.** 269786 of Dec. 26, 1882 to S. H. Hamilton; 631742 of Aug. 22, 1899 to O. L. Gardner; 1248181 of Nov. 27, 1917 to B. W. Sidwell; 1477532 of Dec. 18, 1923 to C. S. Bird; 2075317 of Mar. 30, 1937 to C. A. Upson.

859 [p. 717] **U. S. Pat.** 1479044 of Jan. 1, 1924 to Lester Kirschbraun.

860 [p. 717] **U. S. Pats.** 195483 of Sep. 25, 1877 to J. M. Cobb; 1074829 of Oct. 7, 1913 to C. S. Bird and G. R. Wyman.

861 [p. 717] **U. S. Pats.** 1336403 and 1336404 of Apr. 6, 1920 to H. F. Weiss; 1353619 of Sep. 21, 1920 to H. W. White; 1571667 of Feb. 2, 1926 to O. A. Heppes; 1578663 of Mar. 30, 1926 to H. C. Innes.

862 [p. 717] **U. S. Pats.** 1115593 of Nov. 3, 1914 to J. P. Sexton; Reissue 14148 of Jun. 5, 1916 to J. P. Sexton; 1205360 and 1205399 of Nov. 21, 1916 to J. P. Sexton; 1325883 of Dec. 23, 1919 to J. P. Sexton.

863 [p. 717] **U. S. Pat.** 17983810 of Feb. 24, 1931 to H. L. Levin; **Can. Pat.** 320383 of Mar. 8, 1932 to Flintkote Co.

864 [p. 717] **U. S. Pats.** 1831058 of Nov. 10, 1931 to H. A. Cumfer; 2075373 of Mar. 30, 1937 to John Tomec.

865 [p. 717] **U. S. Pat.** 1821120 of Sep. 1, 1931 to H. M. Spencer.

866 [p. 718] **U. S. Pat.** 1874674 of Aug. 30, 1932 to C. A. Watson.

867 [p. 718] **U. S. Pats.** 1322278 of Nov. 18, 1919 to M. K. Armstrong; 1417836 of May 30, 1922 to Lester Kirschbraun; 1644652 of Oct. 4, 1927 to Lester Kirschbraun.

868 [p. 718] **U. S. Pats.** 985140 of Feb. 28, 1911 to Hedley Button; 1292067 of Jan. 21, 1919 to H. H. Robertson; 1322278 of Nov. 18, 1919 to M. K. Armstrong; 1362888 of Dec. 21, 1920 to T. J. Mullin; 1902298 of Mar. 21, 1933 to H. C. Avery and Lester Kirschbraun;

Can. Pat. 278743 of Mar. 20, 1928 to Flintkote Co.; **Brit. Pats.** 121205 of Dec. 21, 1917 to D. L. Irwin and E. R. James; 138428 of Feb. 11, 1919 to George Harrison.

869 [p. 718] **U. S. Pat.** 1489567 of Apr. 8, 1924 to H. F. Weiss; **Can. Pat.** 303706 of Sep. 9, 1930 to Building Products, Ltd.

870 [p. 718] **U. S. Pat.** 1510233 of Sep. 30, 1924 to O. D. McFarland.

871 [p. 718] **U. S. Pats.** 206850 of Aug. 13, 1878 to D. S. Armstrong; 1473981 of Nov. 13, 1923 to J. W. Wagner.

872 [p. 718] **U. S. Pats.** 1352619 of Sep. 21, 1920 to H. W. White; 1461337 of Jul. 10, 1923 to H. F. Weiss; 2012805 of Aug. 27, 1935 to A. G. Brown and S. E. McPartlin.

873 [p. 718] **U. S. Pat.** 1949087 of Feb. 27, 1934 to M. D. Squiers; **French Pat.** 776920 of Feb. 7, 1935 to Diafan, S. A.

874 [p. 718] **U. S. Pats.** 845920 of Feb. 26, 1909 to E. H. Binns; 891428 of Jun. 23, 1908 to W. H. Latus; 958450 of May 17, 1910 to H. R. Wardell; 999951 of Aug. 8, 1911 to C. S. Bird; 1519280 and 1519281 of Dec. 16, 1924 to Kurt Wandel; 1520284 of Dec. 23, 1924 to G. H. Ellis; 1574886 of Mar. 2, 1926 to O. A. Heppes and W. H. Cady; 1640619 of Aug. 30, 1927 to C. W. Scoggin; 1765796 of Jun. 24, 1930 to Lester Kirschbraun; 1772686 of Aug. 12, 1930 to C. E. Rahr; 1802880 of Apr. 28, 1931 to H. A. Cumfer; 1949255 of Feb. 27, 1934 to C. L. Keller; 2024586 of Jun. 2, 1936 to John Campbell and R. G. Quinn; **Can. Pats.** 133756 of Jun. 13, 1911 to Hermann Schlisske; 251757 of Jul. 14, 1925 to Gardner & Lewis; 256565 of Dec. 22, 1925 to Flintkote Co.; 329434 of Jan. 17, 1933 to Bird & Son, Inc.; 355645 of Jan. 28, 1936 to International Paper Co.; **Brit. Pats.** of 1912 (May 6), 10695 to E. H. Angier; 374298 of Jun. 9, 1932 to Otto Goy; 431162 of Jul. 2, 1935 to John Campbell and R. G. Quinn; **Ger. Pats.** Design 333255 of Feb. 12, 1908 to L. Wunnenberg; Design 415347 of Feb. 26, 1910 to August Preuss; Design 446545 of Nov. 5, 1910 to Bremer Papier und Wellpappenfabrik, A.-G.

875 [p. 718] **U. S. Pats.** 403588 of May 21, 1889 to G. A. Herdman; 527505 of Oct. 16, 1894 to Patrick Norton; 540992 of Jun. 11, 1895 to A. E. Krause; 836157 of Nov. 20, 1906 to P. W. Turner; 1015919 of Jan. 30, 1912 to H. R. Wardell; **Brit. Pat.** 338403 of Dec. 13, 1928 to E. Rudin and Heinrich Kolbrunner; **Ger. Pat.** Design 1338133 of Apr. 26, 1935 to Heinrich Züge.

876 [p. 718] **U. S. Pat.** 1587462 of Jun. 1, 1926 to F. W. Adams.

877 [p. 718] **U. S. Pats.** 1457664 of Jun. 5, 1923 to S. M. Ford; 1926737 of Sep. 12, 1933 to S. J. Blum; **Can. Pats.** 233378 of Aug. 7, 1923 to Barrett Co.; 281500 of Jul. 3, 1928 to C. W. Scoggin and Niels Nissen; **Ger. Pats.** 112629 of May 5, 1899 to A. W. Andernach; 121436 of May 6, 1899 to A. W. Andernach.

878 [p. 718] **U. S. Pats.** 1595673 of Aug. 10, 1926 to G. R. Magney and W. R. Nelson; 1598981 of Sep. 7, 1926 to W. R. Nelson and T. B. Hennessey; 1941769 of Jan. 2, 1934 to G. J. Ward;

879 [p. 718] **U. S. Pat.** 1938351 of Dec. 5, 1933 to E. S. Penn.

880 [p. 718] **U. S. Pat.** 1822632 of Sep. 8, 1931 to H. F. Winkelmann; **Can. Pat.** 324493 of Jul. 26, 1932 to H. F. Winkelmann.

881 [p. 718] **U. S. Pat.** 1152798 of Sep. 7, 1915 to Julius de Long and J. B. d'Homergue.

882 [p. 718] **U. S. Pats.** 1801525 of Apr. 21, 1931 to W. E. Nelson; 1917456 of Jul. 11, 1933 to A. O. Mickelson.

883 [p. 718] **U. S. Pat.** 1945308 of Jan. 30, 1934 to A. C. Fischer.

884 [p. 718] **U. S. Pat.** 2035122 of Mar. 24, 1936 to S. C. Fulton and Vladimir Kalichevsky.

885 [p. 718] **U. S. Pats.** 1883485 and 1883486 of Oct. 18, 1932 to C. J. Beckwith; 1976684 of Oct. 9, 1934 to T. B. Munroe and G. E. Swenson; **Can. Pat.** 324535 of Jul. 26, 1932 to Celotex Co.; **Brit. Pats.** 331561 of Feb. 4, 1929 to Robert Arnot; 372124 of Jun. 4, 1931 to Celotex Co.

886 [p. 718] **Brit. Pat.** 348128 of Dec. 29, 1928 to W. M. Shakespeare.

887 [p. 719] **U. S. Pat.** 1975637 of Oct. 2, 1934 to Dozier Finley.

888 [p. 719] **U. S. Pats.** 868157 of Oct. 15, 1907 to G. F. Bishopric; 875041 of Dec. 31,

1907 to G. F. Bishopric; 1022367 of Apr. 2, 1912 to W. M. Janpole; 1088803 of Mar. 3, 1914 to G. F. Bishopric; **Can. Pats.** 175786 and 175787 of Mar. 20, 1917 to G. H. Ellis; 256565 of Dec. 22, 1925 to Flintkote Co.; **Brit. Pats.** of 1906 (Jul. 5), 15314 and 15314-A to G. F. Bishopric; **Ger. Pat.** 504955 of Jan. 25, 1929 to Ludwig Schwabe.

889 [p. 719] **Ger. Pat.** 754999 of May 27, 1893 to H. I. Noke.

890 [p. 719] **U. S. Pat.** 1876340 of Sep. 6, 1932 to L. S. Odell; **Can. Pat.** 342417 of Jun. 19, 1934 to Building Products, Ltd.

891 [p. 719] See Reference 863.

892 [p. 719] **U. S. Pats.** 1156866 of Oct. 12, 1915 to S. M. Ford; 1208535 of Dec. 12, 1916 to S. M. Ford; 1226650 of May 22, 1917 to S. M. Ford; 1245450 of Nov. 6, 1917 to S. M. Ford; 1249733 of Dec. 11, 1917 to S. M. Ford; 1251879 of Jan. 1, 1918 to S. M. Ford; 1258328 of Mar. 5, 1918 to S. M. Ford; 1266574 of May 21, 1918 to S. M. Ford; 1293072 of Feb. 4, 1919 to S. M. Ford; 1525071 of Feb. 3, 1925 to H. A. Cumfer; **Brit. Pat.** 101147 of Aug. 9, 1915 to S. M. Ford.

893 [p. 719] **U. S. Pat.** 1977046 of Oct. 16, 1934 to H. A. Cumfer.

894 [p. 719] **U. S. Pats.** 825870 of Jul. 10, 1906 to Julius Schirra; 1138069 of May 4, 1915 to Addison Applegate; 1518337 of Dec. 9, 1924 to J. F. Makowski; 1592807 of Jul. 13, 1926 to C. N. Forrest; 1627531 of May 3, 1927 to F. A. Browne; 1700930 of Feb. 5, 1929 to C. N. Forrest; 1707255 of Apr. 2, 1929 to M. S. Darrow; 1952481 of Mar. 27, 1934 to Edward Westberg.

895 [p. 719] **U. S. Pats.** 1802878, 1802879 and 1802880 of Apr. 28, 1931 to H. A. Cumfer; 1972951 of Sep. 11, 1934 to J. H. Plunkett; **Can. Pats.** 274636 of Oct. 11, 1927 to W. E. Nelson; 324175 of Jul. 12, 1932 to Paraffine Cos., Inc.; **Brit. Pat.** 357838 of Nov. 12, 1930 to C. H. Barton; **Ger. Pat.** 442919 of May 15, 1926 to L. Wunsch.

896 [p. 719] **U. S. Pats.** 1776254 of Sep. 23, 1930 to Dozier Finley; 1813081 of Jul. 7, 1931 to J. H. Plunkett; 2018216 of Oct. 22, 1935 to R. S. Maclean.

897 [p. 719] **U. S. Pat.** 1847372 of Mar. 1, 1932 to E. H. Angier.

898 [p. 719] **Ger. Pat.** Design 327350 of Dec. 20, 1907 to Wilhelm Hüter.

899 [p. 719] **U. S. Pat.** 1486535 of Mar. 11, 1924 to T. B. Munroe.

900 [p. 719] **U. S. Pat.** 1993472 of Mar. 5, 1935 to Heinrich Bosari-Fischer.

CHAPTER XXX

1 [p. 720] "Rustless Coatings," by M. P. Wood, 1st Edition, John Wiley & Sons, New York (1904); "The Analysis of Paints and Painting Materials," by H. A. Gardner and J. A. Schaeffer, McGraw-Hill Book Co., New York (1911); "Paints and Varnish," Circular No. 69, Bureau of Standards, Wash., D. C. (Nov. 17, 1917); "The Industrial and Artistic Technology of Paint and Varnish," by A. H. Sabin, 2nd Edition, John Wiley & Sons, Inc., New York (1917); "Varnishes and Their Components," by R. S. Morrell and Henry Frowde; Hodder & Stoughton, London (1923); "Pigmented Bitumastic Compositions," by J. M. Fain and F. D. Snell, *J. Ind. Eng. Chem.*, **26**, 88 (1934); "Bituminous Protective Coatings," by R. Forder, *Paint Manufacture*, 4, 299 (1934); "Varnish Making," by T. H. Barry and G. W. Dunster, Leonard Hill, Ltd., London (1935); "Standard Definitions of Terms Relating to Paint Specifications" (D-16-24), A.S.T.M. Standards 1936, II, 826.

2 [p. 720] **U. S. Pat.** 163223 of May 11, 1875 to J. R. McClintock.

3 [p. 720] **U. S. Pat.** 3598 of May 25, 1844 to Edouard Deutsch.

4 [p. 720] **U. S. Pat.** 89727 of May 4, 1869 to E. V. Audibert.

5 [p. 720] **U. S. Pat.** 67696 of Aug. 13, 1867 to Henry Wurtz.

6 [p. 721] **U. S. Pat.** 178152 of May 30, 1876 to W. P. Jenney.

7 [p. 721] **U. S. Pats.** 616047 of Dec. 13, 1898 to C. E. Anthony; 734482 of Jul. 21, 1903 to S. R. Whitall; 768101 of Aug. 23, 1904 to F. M. Whitall; 984477 of Feb. 14, 1911 to M. D. Griffin; 1302090 of Apr. 29, 1919 to S. H. Reeves; **French Pat.** 381454 of Sep. 2, 1907 to U. S. A. Stinc.

8 [p. 721] **U. S. Pat.** 1009708 of Nov. 21, 1911 to E. W. Enequist.

9 [p. 721] **U. S. Pat.** 190953 of May 22, 1877 to Charles Brown.

10 [p. 721] "Air-Drying Asphaltum Finishes," by E. Assheton, *Paint Manuf.*, **5**, 371 (1935); **U. S. Pats.** 17851 of Jul. 21, 1857 to J. B. Wands; 1016801 of Feb. 6, 1912 to L. S. van Westrum; 1379018 of May 24, 1921 to A. C. Holzapfel; **Brit. Pat.** of 1872 (Aug. 27), 2545 to Campbell Morfit; **Ger. Pat.** 439519 of Jun. 26, 1925 to Ernst Stern.

11 [p. 721] **U. S. Pats.** 30217 of Oct. 2, 1860 to J. P. Gay; 529730 of Nov. 27, 1894 to William Griscom, Jr.; **Ger. Pat.** 386821 of Sep. 6, 1922 to Chem. Labrtorium für Anstreichstoffe, G.m.b.H.

12 [p. 721] **U. S. Pats.** 207096 of Aug. 20, 1878 to M. B. Bailey; 1430083 of Sep. 26, 1922 to A. C. Holzapfel.

13 [p. 721] **U. S. Pat.** 1398084 of Nov. 22, 1921 to Leonard Wickenden; **Ger. Pat.** 248779 of Aug. 25, 1910 to C. F. Boehringer & Söhne; **French Pats.** 714805 and 714806 of Apr. 4, 1931 to New-York-Hamburger Gummiwaren Cie.

14 [p. 721] **U. S. Pat.** 1308575 of Jul. 1, 1919 to Samuel Cabot.

15 [p. 721] **U. S. Pat.** 1462381 of Jul. 17, 1923 to Albert Schmidt; **Ger. Pat.** 248779 of Aug. 23, 1910 to C. F. Boehringer & Söhne.

16 [p. 721] **U. S. Pat.** 1400041 of Dec. 13, 1921 to Carleton Ellis.

17 [p. 721] **U. S. Pat.** 1458493 of Jun. 12, 1923 to Louis Mauerhofer.

18 [p. 721] **Can. Pats.** 336622 and 336623 of Oct. 24, 1933 to Koppers Co.

19 [p. 721] **Brit. Pat.** 320886 of Jul. 19, 1928 to F. W. McRae.

20 [p. 721] "Hardened Rosin and Rosin Esters," by A. Murray, *Chem. Met. Eng.*, **25**, 473 (1921).

21 [p. 721] **Ger. Pat.** 338854 of Dec. 11, 1919 to Chem. Fabr. Flörsheim and Dr. H. Noerdlinger.

22 [p. 721] **U. S. Pats.** 1275778 of Aug. 13, 1918 to F. W. Speer, Jr. and Marc Darrin; 1292907 and 1292908 of Jan. 28, 1919 to F. W. Speer, Jr. and Marc Darrin; 1296776 of Mar. 11, 1919 to Marc Darrin; 1405941 of Feb. 7, 1922 to W. B. Robeson; **Ger. Pat.** 425216 of Dec. 28, 1924 to Karl Rösler.

23 [p. 721] **French Pat.** 509556 of Feb. 9, 1920 to Charles Holzapfel and Paul Walther.

24 [p. 721] **U. S. Pat.** 1992695 of Feb. 26, 1935 to T. C. Ford; **Ger. Pats.** 526783 of Feb. 20, 1927 to Gesellschaft für Strahlungschemie, G.m.b.H.; 549520 of Sep. 4, 1928 to Gesellschaft für Strahlungschemie, G.m.b.H.

25 [p. 721] **Ger. Pats.** 247501 of Sep. 7, 1911 to Janny Goepper and Otto Geiger; 256763 of Jul. 16, 1912 to Janny Goepper and Otto Geiger.

26 [p. 721] **U. S. Pats.** 1934709 of Nov. 7, 1933 to A. M. Alvarado and A. N. Parrett; 1955355 of Apr. 17. 1934 to A. M. Alvarado and A. N. Parrett; 1990474 and 1990475 of Feb. 12, 1935 to A. M. Alvarado.

27 [p. 721] "The Newer Chemistry of Coatings," by Carleton Ellis, *J. Ind. Eng. Chem.*, **25**, 125 (1933).

28 [p. 722] "Standard Specifications for Raw Linseed Oil," (D 234-28), A.S.T.M. Standards 1936, II, 723; "Standard Specifications for Boiled Linseed Oil," (D 260–33), A.S.T.M. Standards 1936, II, 717; "Federal Spec. for Oil; Linseed, Raw," Federal Standard Stock Catalogue, Section IV, (Part 5), JJJ-O-336, Apr. 28, 1931; "Federal Spec. for Oil; Linseed, Boiled," Federal Standard Stock Catalogue, Section IV, (Part 5), JJJ-O-331, May 26, 1931.

29 [p. 722] "Standard Specifications for Raw Tung Oil," (D 12–33), A.S.T.M. Standards 1936, II, 734.

30 [p. 722] "Tentative Specification for Raw Soybean Oil," (D 124–33T), A.S.T.M. Tentative Standards 1936, 583.

31 [p. 722] **U. S. Pats.** 1358226 of Nov. 9, 1920 to Frank McPhillips; 1374161 of Apr. 5, 1921 to C. S. Hathaway.

32 [p. 722] "Standard Specification for Perilla Oil, Raw or Refined," (D 125–23), A.S.T.M. Standards 1936, II, 729.

33 [p. 722] **U. S. Pats.** 283044 of Aug. 14, 1883 to D. B. Turner; 1671229 and 1671230 of May 29, 1928 to C. F. Chona.

34 [p. 722] **U. S. Pats.** 1725791, 1725792, 1725793, 1725794, 1725795, 1725796 and 1725797 of Aug. 27, 1929 to M. T. Harvey.

35 [p. 722] **U. S. Pat.** 2063669 of Dec. 8, 1936 to T. C. Ford.
36 [p. 722] **U. S. Pat.** 1842207 of Jan. 19, 1932 to W. H. O. J. Scheiber.
37 [p. 722] **Ger. Pat.** 93309 of Jul. 10, 1897 to Pierre Janssen.
38 [p. 722] **U. S. Pat.** 1317469 of Sep. 30, 1919 to R. R. Adams; **Can. Pat.** 287921 of Mar. 12, 1929 to Sozol, Ltd.; **Brit. Pat.** 298256 of Jun. 8, 1927 to S. Fowler and E. Edser.
39 [p. 722] **U. S. Pat.** 2071555 of Feb. 23, 1937 to Heinrich Meder; **Ger. Pat.** 38221 of Feb. 17, 1886 to Philipp Cornely.
40 [p. 722] **Ger. Pats.** 565980 of Apr. 3, 1933 to Heinrich Meder; 601248 of Aug. 11, 1934 to Mederit, G.m.b.H.; 601320 of Aug. 13, 1934 to Mederit, G.m.b.H.; 601684 of Aug. 22, 1934 to Mederit, G.m.b.H.
41 [p. 722] "Kautschuklacke und Kautschukkitte," by Otto Merz, Union Deutsche Verlagsgesellachaft, Berlin (1933); "Rubber Paints," by Joseph Rossman, *India Rubber World*, **90**, No. 6, 37; **91**, No. 1, 33 and 42; No. 2, 39; No. 3, 39 (1934); **U. S. Pats.** 22343 of Dec. 21, 1858 to C. A. Bremner; 33194 of Sep. 3, 1861 to Pater Harder; 65179 of Mar. 28, 1867 to O. A. Day and G. W. Bishop; Reissue 5950-C of Jun. 30, 1870 to H. W. Johns; 107382 of Sep. 13, 1870 to H. W. Johns; 109570 of Nov. 19, 1870 to H. J. Ball; 178153 of May 30, 1876 to W. P. Jenney; 346224 of Jul. 27, 1886 to T. C. Roche; 391927 of Oct. 30, 1888 to J. A. Titzel; 1465317 of Aug. 21, 1923 to Domingo Sanguinetti; 1991300 of Feb. 12, 1935 to Gustav Tichy and Heinrich Klas; **Can. Pats.** 12854 of May 21, 1881 to D. D. Pennoyer and C. F. Whitcher; 17741 of Sep. 24, 1883 to Albert Patterson; 22476 of Sep. 17, 1885 to E. A. Horton and C. G. Thomas; 78572 of Dec. 9, 1902 to W. W. Brasington; **Brit. Pats.** of 1843 (Nov. 21), 9952 to Thomas Hancock; of 1913 (Dec. 6), 27128 to A. C. de Caudemberg; 161201 of Jul. 12, 1919 to C. H. Ivinson and G. S. Roberts; 380677 of Sep. 22, 1932 to Maschinenfabrik Oerlikon; 448150 of Oct. 26, 1934 to D. D. Pratt; **Ger. Pats.** 124793 of Dec. 10, 1899 to E. Arthur; 399366 of Jan. 8, 1922 to Domingo Sanguinetti; 545003 of Mar. 26, 1930 to Rudolf Schell; **Swiss Pat.** 180967 of Feb. 17, 1936 to Alphons Wyss.
42 [p. 722] "Bituminous Paints for Submerged Objects," by E. Kindscher, *Farbe u. Lack*, 486 and 497 (1935); **U. S. Pats.** 522312 of Jul. 3, 1894 to A. A. Blandy; 1890723 of Dec. 13, 1932 to Max Deseniss and Andreas Nielsen; **Brit. Pats.** 353311 of Sep. 15, 1930 to Max Deseniss and Andreas Nielsen; 411640 of Apr. 7, 1933 to J. R. Geigy, A.-G.
43 [p. 722] **U. S. Pat.** 2044176 of Jun. 16, 1936 to Leon McCulloch.
44 [p. 722] "The Use of Rubber in Paints," by H. P. Stevens and Noël Heaton, *J. Oil & Colour Chem. Assoc.*, **17**, 8 (1934); "Rubber in Paints and Varnishes," publised by Rubber Growers' Association, London (1935); **British Pats.** 407038 of Jun. 29, 1932 to H. P. Stevens and Noël Heaton; 417912 of Apr. 11, 1933 to H. P. Stevens and Noël Heaton.
45 [p. 722] **U. S. Pat.** 1995957 of Mar. 26, 1935 to H. J. Barrett; **Can. Pat.** 331635 of Apr. 11, 1933 to H. J. Barrett; **Austrian Pat.** 98975 of Aug. 15, 1924 to Hermann Suida.
46 [p. 722] **French Pats.** 760719 and 760720 of Mar. 1, 1934 to Soc. industrielle de produits Chimiques.
47 [p. 722] **U. S. Pat.** 2001430 of May 14, 1935 to J. V. Meigs.
48 [p. 723] **U. S. Pats.** 322804 and 322805 of Jul. 21, 1885 to A. G. Day; 1067536 of Jul. 15, 1913 to Louis Meunier; **Brit. Pats.** of 1880 (Jan. 9), 94 to Richard Condy; of 1888 (Sep. 22), 13721 to W. C. Nangle; 336117 of Oct. 26, 1929 to Craigbank Chemical Co., Ltd. and J. H. Taylor.
49 [p. 723] **U. S. Pat.** 1895320 of Jan. 24, 1933 to J. H. Gravell; 1995954 of Mar. 26, 1935 to H. O. Albrecht; **Can. Pat.** 336447 of Oct. 17, 1933 to H. O. Albrecht; **Brit. Pats.** 355286 of May 20, 1930 to J. H. Gravell; 407008 of Mar. 7, 1934 to E. I. du Pont de Nemours & Co.
50 [p. 723] **U. S. Pats.** 1145186 of Jul. 16, 1915 to Rudolf Eberhard; 1147971 of Jul. 27, 1915 to R. S. Perry.
51 [p. 723] **Ger. Pat.** 397824 of Jan. 14, 1923 to C. F. Beer Söhne.
52 [p. 723] **Brit. Pat.** 355430 of Jun. 30, 1930 to N. V. de Bataafsche Petroleum Maatschappij.
53 [p. 723] **U. S. Pat.** 391927 of Oct. 30, 1888 to J. A. Titzel.
54 [p. 724] "Standard Specifications for Basic Carbonate White Lead" (D 81-34),

A.S.T.M. Standards for 1936, II, 602; " Federal Spec. for White-Lead; Basic-Carbonate, dry, paste-in-oil, and semipaste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-W-251; "Standard Specifications for Basic Sulfate White Lead" (D 82-24), A.S.T.M. Standards 1936, II, 604; "Tentative Specifications for Blue Lead; Basic Sulfate" (D 405-35T), A.S.T.M. Tentative Standards 1936, 577; "Standard Specifications for Zinc Oxide" (D 79-24), A.S.T.M. Standards 1936, II, 606; "Federal Spec. for Zinc-Oxide; dry and paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-Z-301; "Standard Specifications for Leaded Zinc Oxide" (D 80-24), A.S.T.M. Standards 1936, II, 608; "Federal Spec. for Zinc-Oxide; leaded, dry and paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-Z-302; "Standard Specifications for Lithopone" (D 208-26), A.S.T.M. Standards 1936, II, 582; "Standard Specifications for Zinc Sulfide" (D 386-36), A.S.T.M. Standards 1936, II, 610; "Standard Specifications for Zinc Sulfide Pigment (Barium)" (D 385-36), A.S.T.M. Standards 1936, II, 612; "Standard Specifications for Titanium Dioxide" (D 384-36), A.S.T.M. Standards 1936, II, 596; "Standard Specifications for Titanium Barium Pigment" (D 382-35), A.S.T.M. Standards 1936, II, 592; "Standard Specifications for Titanium Calcium Pigment" (D 383-35), A.S.T.M. Standards 1936, II, 594; "Standard Specifications for Mineral Iron Oxide" (D 84-27), A.S.T.M. Standards 1936, II, 578; "Standard Specifications for Red Lead" (D 83-31), A.S.T.M. Standards 1936, II, 590; "Federal Spec. for Red-Lead; dry and paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-R-191; "Standard Specifications for Commercial Para Red" (D 264-28), A.S.T.M. Standards 1936, II, 586; "Standard Specifications for Ocher" (D 85-27), A.S.T.M. Standards 1936, II, 584; "Federal Spec. for Ocher; dry, paste-in-japan, paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-O-111; "Federal Spec. for Chrome Yellow; dry, paste-in-japan, paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-C-291; "Standard Specifications for Chrome Yellow" (D 211-27), A.S.T.M. Standards 1936, II, 576; "Standard Specifications for Chrome Oxide Green," (D 263-28), A.S.T.M. Standards 1936, II, 574; "Standard Specifications for Pure Chrome Green" (D 212-27), A.S.T.M. Standards 1936, II, 570; "Standard Specifications for Reduced Chrome Green" (D 213-27), A.S.T.M. Standards 1936, II, 572; "Federal Spec. for Chrome Green; Oxide," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-C-231 (Jul. 22, 1930); "Federal Spec. for Chrome Green; pure, dry, paste-in-japan; paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-C-236; "Standard Specifications for Prussian Blue" (D 261-28), A.S.T.M. Standards 1936, II, 588; "Federal Spec. for Prussian Blue; dry, paste-in-japan; paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-P-691; "Standard Specifications for Ultramarine Blue" (D 262-28), A.S.T.M. Standards 1936, II, 600; "Federal Spec. for Ultramarine-Blue; dry, paste-in-japan, paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-U-451; "Standard Specifications for Lampblack" (D 209-30), A.S.T.M. Standards 1936, II, 580; "Federal Spec. for Lampblack; dry, paste-in-japan, paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-L-71; "Standard Specifications for Bone Black" (D 210-30), A.S.T.M. Standards 1936, II, 568; "Federal Spec. for Boneblack; dry, paste-in-japan, paste-in-oil," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-B-601; "Standard Specifications for Aluminum Powder for Paints (Aluminum Bronze Powder)" (D 266-31), A.S.T.M. Standards 1936, II, 565; "Standard Specifications for Gold Bronze Powder" (D 267-31), A.S.T. M. Standards 1936, II, 567; "Standard Method of Test for Determination of Polishing Lubricant in Aluminum Powder for Paints (Aluminum Bronze Powder)" (D 306-31), A.S.T.M. Standards 1936, II, 616.

55 [p. 724] **Ger. Pat.** 357611 of Aug. 26, 1918 to A. Blumenthal & Co., Chem. Fabrik.

56 [p. 724] **U. S. Pat.** 240899 of May 3, 1881 to J. L. Fauss; **Can. Pat.** 23425 of Feb. 15, 1886 to Ferronite Mfg. Co.; **Brit. Pat.** 223486 of Mar. 6, 1924 to A. A. Gorvan; **Ger. Pat.** 163002 of Nov. 6, 1904 to F. C. Matthies et Cie.

57 [p. 724] **U. S. Pats.** 79645 of Jul. 7, 1868 to L. D. Ferguson; 231540 of Aug. 24, 1880 to John Collins; 1379542 of May 24, 1921 to T. J. Ethridge; **Can. Pat.** 3786 of Aug. 24, 1874 to Terrence Sparham.

58 [p. 724] **U. S. Pat.** 1145782 of Jul. 6, 1915 to J. H. Matthes.

59 [p. 724] "The Selection and Application of Bituminous Materials for Paints," by Paul Walther, *Farben-Ztg.*, 40, 1196 (1935).

60 [p. 724] **Ger. Pat.** 24231 of Sep. 14, 1883 to Emil Heuser.

61 [p. 724] "Aluminum Bronze Powder and Aluminum Paint," by J. D. Edwards, The Chemical Catalog Co., Inc., New York (1927); "Protection of Metal Structures with Bituminous Paints," by J. Roux, Rev. Aluminum, 12, 2745 (1935); "Pigmentation of Bituminous Paints," by W. Ludwig, *Farben-Ztg.*, 41, 1282 (1936); "Aluminum Paint and Powder," by J. D. Edwards, 2nd Edition, Revised, The Chemical Catalog Co., Inc., New York (1936); **U. S. Pats.** 1568215 of Jan. 5, 1926 to C. S. Fleming; 1765676 of Jun. 24, 1930 to L. C. Jones; 1932502 of Oct. 31, 1933 to R. A. Altenhof; 1986591 of Jan. 1, 1935 to E. B. Meyer; 1992695 of Feb. 26, 1935 to T. C. Ford; **Can. Pat.** 263415 of Aug. 10, 1926 to Paraffine Cos., Inc.; 351480 of Jul. 9, 1935 to American Asphalt Paint Co.; 358044 of May 19, 1936 to E. B. Meyer; **Brit. Pat.** 291486 of Jan. 28, 1927 to S. A. McMinn; **Ger. Pats.** 382519 of Jul. 16, 1921 to Katharina Wickel; 579196 of Jun. 22, 1933 to Friedrich Mitterberger; **French Pat.** 738198 of Jun. 6, 1932 to André Diebold.

62 [p. 724] "Federal Spec. for Aluminum-powder; (for) Paints (Aluminum-Bronze-Powder)," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-A-476, Nov. 15, 1932.

63 [p. 724] **Ger. Pat.** 368236 of Dec. 24, 1920 to Lack- und Farbenfabriken Max Rogler.

64 [p. 725] "Tentative Specification for Petroleum Spirits (Mineral Spirits)," (D 235–34T), A.S.T.M. Tentative Standards 1936, 581; "Federal Spec. for Gasoline, Motor, United States Government," Federal Standard Stock Catalogue, Section IV, (Part 5), VV-G-101, Jul. 21, 1931; "Federal Spec. for Thinner; Paint, Volatile Mineral Spirits," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-T-291, March 31, 1931; "Federal Spec. for Thinner; Paint, (for) Semipaste Paints," Federal Stock Catalogue, Section IV, (Part 5), TT-T-271, Mar. 31, 1931.

65 [p. 725] "Federal Spec. for Ether; Petroleum," Federal Standard Stock Catalogue, Section IV, (Part 5), O-E-751, Jan. 6, 1931.

66 [p. 725] **Brit. Pats.** 412495 of Dec. 8, 1933 to Alexander Wacker Gesellschaft für elektrochem. Industrie, G.m.b.H.; 432406 of Nov. 22, 1934 to Alexander Wacker Gesellschaft für elektrochem. Industrie G.m.b.H.; **French Pat.** 773577 of Nov. 21, 1934 to Alexander Wacker Gesellschaft für elektrochem. Industrie, G.m.b.H.

67 [p. 725] Barrett Co., with additions; "Solvents from the Gas Industry," by C. R. Downs, *J. Soc. Chem. Ind.*, **51**, 28 and 45 (1932).

68 [p. 725] "Standard Specifications for Industrial 90 per cent Benzene (Benzol)," (D 361–36), A.S.T.M. Standards 1936, II, 788.

69 [p. 725] "Standard Specification for Industrial Pure Toluene (Toluol)," (D 362–36), A.S.T.M. Standards 1936, II, 808.

70 [p. 725] "Standard Specification for Industrial Xylene (Xylol), Solvent Naphtha," (D 364–36), A.S.T.M. Standards 1936, II, 812.

71 [p. 725] "Standard Specifications for Spirits of Turpentine," (D 13–34), A.S.T.M. Standards, 1936, II, 742; "Standard Methods of Sampling and Testing Turpentine," (D 233–36), A.S.T.M. Standards 1936, II, 744; "Federal Spec. for Turpentine; (for) Paint, Type I, (Gum-Spirits of Turpentine and Steam-Distilled Wood Turpentine)," Federal Standard Stock Catalogue, Section IV, (Part 5), LLL-T-791a, Jul. 26, 1932 (Bureau of Standards Circular C-86); "Federal Spec. for Turpentine; (for) Paint, Type II," Federal Standard Stock Catalogue, Section IV, (Part 5), LLL-T-792, Jun. 6, 1933; **U. S. Pats.** 162394 of Apr. 20, 1875 to A. K. Lee; Reissue 8921 of Oct. 7, 1879 to A. K. Lee.

72 [p. 725] "Standard Specifications for Acetone," (D 329–33), A.S.T.M. Standards 1936, II, 784; "Federal Spec. for Acetone," Federal Standard Stock Catalogue, Section IV, (Part 5), O-A-51a, Aug. 1, 1933.

73 [p. 725] **U. S. Pats.** 135879 of Feb. 18, 1873 to Peter Barthel; 338868 of Mar. 30, 1886 to T. J. Pearce and M. W. Beardsley; 348993, 348994, 348995 and 348996 of Sep. 14, 1886 to T. J. Pearce and M. W. Beardsley; **Ger. Pat.** 63437 of Nov. 21, 1891 to Ludwig Haarmann.

74 [p. 725]. **U. S. Pat.** 1535999 of Apr. 28, 1925 to A. G. Harrington; **Ger. Pat.** 302667 of Jun. 5, 1914 to O. Weber.

75 [p. 725] **U. S. Pats.** 835113 of Nov. 6, 1906 to S. G. Penney; 903275 of Nov. 10, 1908 to W. F. Doerflinger and L. H. Buck; 1156119 of Oct. 12, 1915 to George Whigelt; 1204056 of Nov. 7, 1916 to J. O. Parsons; **Can. Pat.** 333224 of Jun. 13, 1933 to Carbide & Carbon Chemicals Corp.; **Brit. Pat.** 363698 of Jul. 21, 1930 to Anton Breuer; **Ger. Pat.** 312064 of May 13, 1919 to Eduard Herzinger and Edmund Naylor.

76 [p. 725] "Flash, Fire and Explosion Tests on Mixtures of Carbon Tetrachloride and Naphtha," by E. A. Barrier, *J. Ind. Eng. Chem.*, **2**, 16 (1910); "Non-flammable Mixtures of Organic Solvents," by A. F. Sievers and J. D. McIntyre, *Chem. Met. Eng.*, **26**, 603 (1922); **U. S. Pats.** 1989478 of Jan. 29, 1935 to J. J. Grebe, S. M. Stoesser and L. E. Mills; 2027686 of Jan. 14, 1936 to Paul Friedrich; **Brit. Pat.** 373769 of May 14, 1931 to Anton Breuer; **Ger. Pat.** Appl. B–148940 of Mar. 13, 1931 to Anton Breuer.

77 [p. 725] **Brit. Pat.** 231411 of Apr. 25, 1924 to Paul Lechler.

78 [p. 725] **Ger. Pat.** 291461 of Jan. 10, 1915 to O. Heublein.

79 [p. 725] **U. S. Pat.** 1255618 of Feb. 5, 1918 to Georges Lépine.

80 [p. 725] **U. S. Pat.** 1155130 of Sep. 28, 1915 to S. R. Church; **French Pat.** 756481 of Dec. 11, 1933 to Paul Friedrich.

81 [p. 725] **U. S. Pat.** 1635567 of Jul. 12, 1927 to H. C. P. Weber.

82 [p. 725] **U. S. Pat.** 1635567 of Jul. 12, 1927 to H. C. P. Weber.

83 [p. 725] **Brit. Pat.** 358974 of Jul. 13, 1929 to C. A. Agthe.

84 [p. 725] **Ger. Pats.** 606673 of Dec. 10, 1932 to Alexander Wacker Ges. für elektrochemische Industrie, G.m.b.H.; 615757 of Dec. 21, 1933 to Alexander Wacker Ges. für elektrochemische Industrie, G.m.b.H.; **French Pats.** 761750 of Mar. 26, 1934 to Alexander Wacker Ges. für elektrochemische Industrie, G.m.b.H.; 780112 of Apr. 19, 1935 to Alexander Wacker Ges. für elektrochemische Industrie, G.m.b.H.

85 [p. 725] **Ger. Pat.** 575921 of May 5, 1933 to Paul Lechler.

86 [p. 726] **Ger. Pat.** Appl. S–110330 of Jul. 25, 1933 to S. A. John Cockerill.

87 [p. 726] **U. S. Pat.** 1305790 of Jun. 3, 1919 to C. N. Forrest and J. V. Meigs.

88 [p. 726] Barrett Co., with additions.

89 [p. 726] "Evaporation Rates of Organic Liquids," by H. E. Hofmann, *J. Ind. Eng. Chem.*, **24**, 135 (1932); "Nitrocellulose Lacquers—Rate of Evaporation of Liquids," by F. A. Bent and S. N. Wik, *J. Ind. Eng. Chem.*, **28**, 312 (1936).

90 [p. 727] **U. S. Pats.** 1803637 of May 5, 1931 to I. J. Novak; 1925085 of Sep. 5, 1933 to I. J. Novak; 2066289 of Dec. 29, 1936 to William Hassard; **Can. Pat.** 321209 of Apr. 5, 1932 to Canadian Raybestos Co., Ltd.; **Brit. Pat.** 369396 of Mar. 20, 1931 to I. J. Novak.

91 [p. 728] "The Jellying of Asphalt Paints," by H. C. Fisher, *J. Ind. Eng. Chem.*, **16**, 509 (1924).

92 [p. 729] **French Pats.** 38505 of Dec. 23, 1907 to M. Duprè and Severin Icard; 413063 of Feb. 26, 1910 to Soc. Debauge et Cie.

93 [p. 729] **U. S. Pats.** 1340855 of May 18, 1920 to J. M. Weiss; 1355102 of Oct. 5, 1920 to F. W. Yeager; 2069927 of Feb. 9, 1937 to E. O. Rhodes; 2069929 of Feb. 9, 1937 to J. H. Swanberg; **Can. Pat.** 218878 of May 23, 1922 to Barrett Co.

94 [p. 729] **U. S. Pat.** 1275778 of Aug. 13, 1918 to F. W. Sperr, Jr.; **Can. Pat.** 188272 of Jan. 14, 1919 to F.W. Sperr, Jr.

95 [p. 729] **U. S. Pats.** 1292907 and 1292908 of Jan. 28, 1919 to F. W. Sperr, Jr. and Marc Darrin; 1296776 of Mar. 11, 1919 to Marc Darrin; **Can. Pats.** 184033 of Apr. 30, 1918 to F. W. Sperr, Jr.; 195043 of Dec. 16, 1919 to Marc Darrin.

96 [p. 729] **Brit. Pat.** 425214 of Jun. 1, 1933 to Paul Friedrich; **Ger. Pat.** 567252 of Sep. 11, 1931 to Paul Friedrich.

97 [p. 729] **U. S. Pat.** 1355099 of Oct. 5, 1920 to J. M. Weiss.

98 [p. 729] **Ger. Pat.** 282712 of Jun. 30, 1914 to Mittelrheinische Teerprodukten- und Dachpappenfabrik A. W. Andernach.

99 [p. 730] **U. S. Pat.** 1783702 of Dec. 2, 1930 to M. M. Connolly.

100 [p. 730] **U. S. Pat.** 188646 of Mar. 20, 1877 to A. K. Lee.

101 [p. 731] "Tests of Integral and Surface Waterproofings for Concrete," by C. H. Jumper, *Bureau of Standards J. of Research*, 7, 1147 (1931); G. W. Washa, *Am. Concrete*

Inst. J., **5**, 1 (1933); "Waterproofers," by H. M. Llewellyn, *Chemistry & Industry*, 929 (1934).

102 [p. 732] "Exterior Waterproofing Materials for Masonry," by D. W. Kessler, *J. Research, National Bureau of Standards*, **14**, 317 (1935).

103 [p. 732] "Exposure Texts on Colorless Waterproofing Materials," by D. W. Kessler, Technologic Paper No. 248, Bureau of Standards, Wash., D. C. (Jan. 7, 1924).

104 [p. 732] **U. S. Pat.** 1292964 of Jan. 28, 1919 to W. A. Ruddell.

105 [p. 732] **U. S. Pat.** 1958397 of May 8, 1934 to E. W. Scripture, Jr.

106 [p. 732] **U. S. Pat.** 1867421 of Jul. 12, 1932 to W. H. Rowan and H. K. Buckner.

107 [p. 732] **U. S. Pat.** 1396674 of Nov. 8, 1921 to F. G. White.

108 [p. 732] **U. S. Pat.** 1282460 of Oct. 22, 1918 to C. H. Parkin.

109 [p. 732] **U. S. Pats.** 1505112 of Aug. 19, 1924 to Joseph von Vass; 1736768 of Nov. 26, 1929 to C. D. Boynton; 1805632 of May 19, 1931 to K. A. Milar; 1925214 of Sep. 5, 1933 to W. H. Storm; 1989696 of Feb. 5, 1935 to R. E. Kelley; 2032789 of Mar. 3, 1936 to Svend Bramsen; 2036837 of Apr. 7, 1936 to R. M. Stephenson; **Ger. Pat.** Design 1230206 of Jan. 22, 1931 to Alfred Ott.

110 [p. 733] **Ger. Pat.** 132661 of Jul. 1, 1902 to Eduard Oehring.

111 [p. 733] "Protection of Concrete against Alkali," by E. C. E. Lord, *Public Roads*, **5**, 23 (1924); **6**, 251 (1926); **8**, 105 (1927); **12**, 109 (1931).

112 [p. 733] **U. S. Pat.** 1693718 of Dec. 4, 1928 to E. C. E. Lord.

113 [p. 733] **U. S. Pat.** 1671995 of Jun. 5, 1928 to W. K. Nelson; **Ger. Pat.** 66892 of Feb. 2, 1892 to John Robson.

114 [p. 733] **Ger. Pat.** 222768 of Mar. 22, 1908 to Gustav Schmidt & Co.

115 [p. 733] **Brit. Pat.** of 1910 (Jul. 7), 16264 to T. D. Kelly; **Ger. Pats.** 340580 of Apr. 17, 1917 to M. O. Höchtel and E. Höchtel; 341742 of Jan. 23, 1918 to M. O. Höchtel and E. Höchtel.

116 [p. 733] **U. S. Pat.** 1932502 of Oct. 31, 1933 to R. A. Altenhof.

117 [p. 733] **U. S. Pats.** 825870 of Jul. 10, 1906 to Julius Schirra; 1282460 of Oct. 22, 1918 to C. H. Parkin; 1848014 of Mar. 1, 1932 to A. C. Horn; 1988450 of Jan. 22, 1935 to R. E. Kelley.

118 [p. 733] **U. S. Pat.** 1262344 of Apr. 9, 1918 to A. C. Holzapfel; **Can. Pat.** 351002 of Jun. 18, 1935 to K. E. R. Rodwell.

119 [p. 733] **French Pat.** 782876 of Jun. 14, 1935 to O. A. Hubert and Pierre Lambert.

120 [p. 733] **U. S. Pats.** 1602105 of Oct. 5, 1926 to P. L. Geer and H. F. Wiggins; 1796269 of Mar. 10, 1931 to Joseph Rose; 1962483 of Jun. 12, 1934 to Karl Daimler and Paul Kurzenberger; 2061098 of Nov. 17, 1936 to A. A. Johnson; **Can. Pat.** 328616 of Dec. 20, 1932 to Joseph Rose; **Brit. Pat.** 373609 of Dec. 24, 1931 to Joseph Rose; 425848 of Dec. 12, 1933 to K. E. H. Rodwell; **French Pat.** 779945 of Apr. 16, 1935 to K. E. H. Rodwell.

121 [p. 733] **U. S. Pat.** 705625 of Jul. 29, 1902 to W. A. Tucker.

122 [p. 733] **U. S. Pats.** 909167, 909168 and 909169 of Jan. 12, 1909 to T. A. Edison.

123 [p. 733] **Ger. Pat.** 6215 of Sep. 18, 1878 to David Urner.

124 [p. 734] "Federal Spec. for Asphalt-Primer; (for) Roofing and Waterproofing," SS-A-701, Federal Standard Stock Catalogue, Section IV, (Part 5), Aug. 1, 1933.

125 [p. 734] "Vergleichende Versuche mit Rohteer, destilliertem und präpariertem Teer und Prüfungen von Roh- und Dachpappe," Staatlichen Materialprüfungsamtes in Berlin-Lichterfeld, Verlag von Wilhelm Knapp, Halle a-S., Germany (1924).

126 [p. 734] **Ger. Pats.** 10685 of Nov. 13, 1879 to Emil Dreyssig; 120785 of May 17, 1901 to H. O. Köhler; 638589 of Apr. 9, 1931 to A. T. Arnot.

127 [p. 734] **Ger. Pat.** 73122 of Jan. 17, 1893 to C. Richard.

128 [p. 734] **Ger. Pat.** 14372 of Aug. 21, 1880 to Wilhelm Thörner.

129 [p. 734] **Ger. Pat.** 321213 of Jun. 21, 1918 to Deutsche Succol, G.m.b.H.

130 [p. 734] **Ger. Pat.** 61555 of May 21, 1891 to Georg Friedrich & Co.

131 [p. 734] **Ger. Pat.** 222768 of Mar. 22, 1908 to Gustav Schmidt & Co.

132 [p. 734] **U. S. Pat.** 706119 of Aug. 5, 1902 to J. C. Shaw, W. J. Shaw and R. E. Shaw.

133 [p. 734] **U. S. Pats.** 135865 of Feb. 11, 1873 to W. A. Torrey; 423047 of Mar. 11,

1890 to W. B. Grover; 713846 of Nov. 18, 1902 to W. W. Brasington; **Ger. Pat.** 399366 of Jan. 8, 1922 to Domingo Sanguinetti.

134 [p. 734] **U. S. Pat.** 707677 of Aug. 26, 1902 to F. H. Crass.

135 [p. 734] **U. S. Pat.** 733597 of Jul. 14, 1903 to G. E. Reynolds.

136 [p. 734] **U. S. Pat.** 707710 of Aug. 26, 1902 to F. A. Mitchell.

137 [p. 734] **U. S. Pats.** 207096 of Aug. 20, 1878 to M. B. Bailey; 1430083 of Sep. 26, 1922 to A. C. Holzapfel.

138 [p. 735] **U. S. Pat.** 106327 of Aug. 16, 1870 to W. B. Coates.

139 [p. 735] **U. S. Pat.** 45275 of Nov. 29, 1864 to A. Robinson.

140 [p. 735] **Ger. Pat.** 229181 of Oct. 30, 1907 to Hermann Engelhardt.

141 [p. 735] **U. S. Pats.** 291600 of Jan. 8, 1884 to Josiah Jowitt; 385057 of Jun. 36, 1888 to Alexander Jones; 791312 of May 30, 1905 to C. S. Bird; **Ger. Pat.** 394707 of Jun. 28, 1922 to A. E. A. Schwarze.

142 [p. 736] **Austrian Pat.** 12296 of Jun. 25, 1903 to Heilpern & Haas.

143 [p. 736] **Ger. Pat.** 160865 of Aug. 14, 1901 to Otto Thiele.

144 [p. 736] **Ger. Pat.** 411114 of Jul. 26, 1922 to Johann Kirschbraun.

145 [p. 736] **Ger. Pat.** 6215 of Sep. 18, 1878 to David Urner.

146 [p. 736] **Ger. Pat.** 70852 of Mar. 26, 1892 to Hermann Gentzen.

147 [p. 736] **Ger. Pat.** 447470 of Feb. 27, 1925 to Haba, G.m.b.H.

148 [p. 736] **U. S. Pats.** 734482 of Jul. 21, 1903 to S. R. Whitall; 768101 of Aug. 23, 1904 to F. M. Whitall; 824898 of Jul. 3, 1906 to Herbert Abraham; 936203 of Oct. 5, 1909 to J. A. West; 1379018 of May 24, 1921 to A. C. Holzapfel; 1430083 of Sep. 26, 1922 to A. C. Holzapfel; **Can. Pat.** 100702 of Aug. 28, 1906 to Standard Paint Co.; **Brit. Pat.** of 1906 (Mar. 23), 7073 to W. Clark.

149 [p. 736] "Federal Spec. for Roof-Coating; Asphalt, Fibrous," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-451, Aug 1, 1933; **Ger. Pat.** 160660 of Dec. 6, 1902 to Heilpern & Haas; **Austrian Pat.** 15127 of Oct. 1, 1903 to Heilpern & Haas.

150 [p. 736] **U. S. Pat.** 1410790 of Mar. 28, 1922 to J. H. Young.

151 [p. 736] **U. S. Pat.** 1536549 of May 5, 1925 to J. H. Young.

152 [p. 736] "Painting on Bitumen," by H. Banc, Peint., Pig., Ver, 13, 142 (1936); **U. S. Pats.** 1539512 of May 26, 1925 to Thomas Robinson; 1631604 of Jun. 7, 1927 to Thomas Robinson.

153 [p. 736] **Ger. Pat.** 368236 of Dec. 24, 1920 to Lack- u. Farbenfabriken Max Rogler.

154 [p. 736] **U. S. Pat.** 1684593 of Sep. 18, 1928 to F. W. McRae.

155 [p. 736] **Ger. Pat.** Appl. C-31135 of Sep. 20, 1921 to Chemische Werke Dotzheim (withdrawn).

156 [p. 736] **U. S. Pat.** 109757 of Nov. 29, 1870 to T. C. Rice.

157 [p. 736] **U. S. Pats.** 300729 of Jun. 17, 1884 to O. F. Parsons; 301742 of Jul. 8, 1884 to S. N. Metzler; 527068 of Oct. 9, 1894 to D. L. Means; 1791455 of Feb. 3, 1931 to J. D. Walston; **Ger. Pat.** 18987 of Dec. 22, 1881 to Arthur Siebel.

158 [p. 736] **Ger. Pat.** 14958 of Dec. 30, 1880 to C. Richard.

159 [p. 737] **Ger. Pat.** 542777 of Sep. 5, 1930 to Bakelite Gesellschaft, G.m.b.H.

160 [p. 737] **Ger. Pats.** 298129 of Sep. 20, 1916 to Rudolf Plönnis; 405299 of Sep. 13, 1922 to Preszburger Dachpappen-, Holzcement-, Asphalt- und Isolierplatten-Fabrik, K. C. Menzel & Paul Meyersberg.

161 [p. 737] "Federal Spec. for Roof-Coating; Asphalt, Brushing Consistency," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-R-451, Aug. 1, 1933.

162 [p. 737] "Acid-proof Coatings for Concrete Surfaces," Letter Circular 42, Revised, Bureau of Standards, Wash., D. C. (Feb. 12, 1923).

163 [p. 738] "Spec. for Paint, Acid-Proof, Black for Ammunition," 3-106C, issued by the U. S. Army.

164 [p. 739] **U. S. Pats.** 1095988 of May 5, 1914 to Carl Roth; 1558082 of Oct. 20, 1925 to Daniel Gardner; **Brit. Pat.** 195995 of Oct. 11, 1921 to Daniel Gardner; **Ger. Pat.** 251710 of Feb. 9, 1912 to Carl Roth.

165 [p. 739] **U. S. Pat.** 1458493 of Jun. 12, 1923 to Louis Mauerhofer.

166 [p. 739] **Ger. Pat.** 480339 of Jan. 1, 1928 to Karl Tietz.

167 [p. 739] **U. S. Pats.** 3598 of May 25, 1844 to Edouard Deutsch; 16457 of Jan. 20, 1857 to R. H. Smith; 16739 of Mar. 3, 1857 to C. R. Milks; 16770 of Mar. 3, 1857 to N. A. Dyar; 17883 of Jul. 28, 1857 to S. K. Lighter and J. A. Morrell; 19695 of Mar. 23, 1858 to Robert Glennon; 19712 of Mar. 23, 1858 to B. L. Prime; 19778 of Mar. 30, 1858 to J. B. Hyde; 20173 of May 4, 1858 to Richard Simons; 21927 of Oct. 26, 1858 to Josee Johnson; 22343 of Dec. 21, 1858 to C. A. Bremner; 22563 of Jan. 11, 1859 to Henry Lester; 23108 of Mar. 1, 1859 to O. S. Oaks; 24072 of May 17, 1859 to J. C. Worth; 25182 of Aug. 23, 1859 to Joseph Ditto and Henry Van Bergen; 25324 of Sep. 6, 1859 to M. D. Du Bois; 32290 of May 14, 1861 to C. C. Hoff; 33194 of Sep. 3, 1861 to Peter Harder; 34251 of Jan. 28, 1861 to Hiram Grant; 35464 of Jun. 3, 1862 to Jonathan Mosteller; 47275 of Apr. 18, 1865 to N. E. Blake; 53716 of Apr. 3, 1866 to W. B. Wheeler; 54426 of May 1, 1866 to C. D. Smith; 60037 of Nov. 27, 1866 to S. B. Moore; 61036 of Jan. 8, 1867 to C. B. Allen; 61427 of Jan. 22, 1867 to Cyrus Hill; 65179 of May 28, 1867 to O. A. Day and G. W. Bishop; 69612 of Oct. 8, 1867 to O. M. Bartholomew; 69917 of Oct. 15, 1867 to A. P. Jackson; 71597 of Dec. 3, 1867 to Samuel Fields; 74343 of Feb. 11, 1868 to R. C. Graves; 76773 of Apr. 14, 1868 to H. W. Johns; 79645 of Jul. 7, 1868 to L. D. Ferguson; 80029 of Jul. 14, 1868 to H. M. Teasdale; 82507 of Sep. 29, 1868 to T. E. Wood; 82529 of Sep. 29, 1868 to J. A. Jones; 84119 of Nov. 17, 1868 to C. F. Hinman; 84820 of Dec. 8, 1868 to Marien Gould; 84856 of Dec. 15, 1868 to Bork Capron; 85871 of Jan. 12, 1869 to W. M. Stuart and A. J. Chapman; 86018 of Jan. 19, 1869 to James Judge; 87050 of Feb. 16, 1869 to L. B. Joy; 95378 of Sep. 28, 1869 to E. W. Ranney; 98911 of Jan. 18, 1870 to R. O. Benton; 99088 of Jan. 25, 1870 to C. F. Hinman; 101071 of Mar. 22, 1870 to T. E. Wood; 107382 of Sep. 13, 1870 to W. H. Jones; 190002 of Nov. 8, 1870 to J. P. Godfrey; 109246 of Nov. 15, 1870 to Oliver Porter; 109757 of Nov. 19, 1870 to T. C. Rice; 136722 of Mar. 11, 1873 to S. C. Hogue; 148910 of Mar. 24, 1874 to Alexander Wilkinson; 153253 of Jul. 21, 1874 to Thomas Heap; 163373 of May 18, 1875 to W. S. Gray and F. W. Gray; 188648 of Mar. 20, 1877 to John Collins; 207096 of Aug. 20, 1878 to M. B. Bailey; 225094 of Mar. 2, 1880 to G. W. Bender; 225679 of Mar. 16, 1880 to A. T. Woodward; 231540 of Aug. 24, 1880 to John Collins; 235365 of Dec. 14, 1880 to A. K. Lee; 237017 of Jan. 25, 1881 to J. F. Hoffman; 240899 of May 3, 1881 to J. L. Fauss; 241803 of May 24, 1881 to A. F. Hawthorn and G. W. Hawthorn; 243990 of Jul. 5, 1881 to J. C. Smith; 281999 of Jul. 24, 1883 to J. B. Hyde; 283200 of Aug. 14, 1883 to H. R. Brissett and John Howe; Reissues 10403 and 10404 of Nov. 6, 1883 to J. B. Hyde; 298072 of May 6, 1884 to D. H. Dorsett; 338868 of Mar. 30, 1886 to T. J. Pearce and M. W. Beardsley; 339777 of Apr. 13, 1886 to John Howe; 348993, 348994 and 348995 of Sep. 14, 1886 to T. J. Pearce and M. W. Beardsley; 369301 of Aug. 3, 1887 to G. W. Swan; 391927 of Oct. 30, 1888 to J. A. Titzel; 394396 of Dec. 11, 1888 to F. M. Reed; 452086 of May 12, 1891 to W. S. Smith; 527068 of Oct. 9, 1894 to D. L. Means; 530898 of Dec. 11, 1894 to Edward Nolon; 531969 of Jan. 1, 1895 to B. F. Rook; 542086 of Jul. 2, 1895 to W. T. Leathers; 547295 of Oct. 1, 1895 to Frederick Salathé; 555221 of Feb. 25, 1896 to Theodore Mott; 566183 of Aug. 18, 1896 to Pierre Janssen; 601910 of Apr. 5, 1898 to Taylor Stephenson; 607348 of Jul. 12, 1898 to Charles Day; 615079 of Nov. 29, 1898 to D. F. Lucas; 651582 of Jun. 12, 1900 to H. Schneider; 701743 of Jun. 3, 1902 to T. L. Lee; 704959 of Jul. 15, 1902 to G. W. Doore and A. J. Doore; 714521 of Nov. 25, 1902 to Christian Schallberger; 715751 of Dec. 16, 1902 to J. W. Brown; 734482 of Jul. 21, 1903 to S. R. Whitall; 763746 of Jun. 28, 1904 to A. L. Ginter; 768101 of Aug. 23, 1904 to F. M. Whitall; 791538 of Jun. 6, 1905 to C. G. Coburn; 795116 of Jul. 18, 1905 to R. H. Goddin; 822946 and 822947 of Jun. 12, 1906 to P. R. Herschman; 826180 of Jul. 17, 1906 to C. C. May; 841573 of Jan. 15, 1907 ro J. A. Ryan; 853354 of May 14, 1907 to August Gross and A. C. Horn; 858824 of Jul. 2, 1907 to J. C. Perry; 862454 of Aug. 6, 1907 to J. A. Dubbs; 862692 of Aug. 6, 1907 to G. W. Allen; 936203 of Oct. 5, 1909 to J. A. West; 967337 of Aug. 16, 1910 to D. T. Day; 981916 of Jan. 17, 1911 to J. T. Couse; 984477 of Feb. 14, 1911 to M. D. Griffin; 1016801 of Feb. 6, 1912 to L. S. Van Westrum; 1185722 of Jun. 6, 1916 to R. P. Safford; 1200664 of Oct. 10, 1916 to R. S. Sollander; 1205845 of Nov. 21, 1916 to H. A. Biggar; 1226889 of May 22, 1917 to Jacques Jacobs; 1254788 of

Jan. 29, 1918 to Louis Flecchia; 1260012 of Mar. 19, 1918 to F. W. Mueller; 1278290 of Sep. 10, 1918 to W. I. Baker; 1283913 of Nov. 5, 1918 to Weller Rodenberger; 1328282 of Jan. 20, 1920 to A. C. Holzapfel; 1344861 of Jun. 29, 1920 to Daniel Campbell; 1349601 of Aug. 17, 1920 to H. L. Buckner and Edmund Burke; 1353239 of Sep. 21, 1920 to C A. Honig; 1358226 of Nov. 9, 1920 to Frank McPhillips; 1362241 of Dec. 14, 1920 to T. J. Duffin; 1374161 of Apr. 5, 1921 to C. S. Hathaway; 1379542 of May 24, 1921 to T. J. Ethridge; 1381112 of Jun. 14, 1921 to W. R. Emig; 1396674 of Nov. 8, 1921 to F. G. White; 1401261 of Dec. 27, 1921 to D. A. Jerone; 1402500 of Jan. 3, 1922 to J. W. Howland; 1404501 of Jan. 24, 1922 to W. V. Watson; 1405941 of Feb. 7, 1922 to W. B. Robeson; 1428273 of Sep. 5, 1922 to W. A. Collings; 1430142 of Sep. 26, 1922 to Charles Appleton and Forbes McRae; 1658540 of Feb. 7, 1928 to George Sutherland; 1684593 of Sep. 18, 1928 to F. W. McRae; 1791455 of Feb. 3, 1931 to J. D. Walston; 1900211 and 1900212 of Mar. 7, 1933 to V. L. Watson; 1957179 of May 1, 1934 to K. A. Milar; **Can. Pats.** 2530 of Jul. 24, 1873 to W. H. Foran; 5297 and 5299 of Oct. 30, 1875 to J. B. Slichter; 17585 of Sep. 1, 1883 to L. D. Mott; 18282 of Dec. 12, 1883 to P. A. Way; 18380 of Jan. 3, 1884 to Albert Sorg and F. D. Phillips; 19802 of Jul. 16, 1884 to George Learmouth and C. H. McCarger; 19868 of Jul. 30, 1884 to Thomas Head; 20382 of Oct. 15, 1884 to H. C. Petty; 20733 of Dec. 15, 1884 to C. M. Lewis; 21116 of Feb. 19, 1885 to W. H. Wilbur and P. P. Seeber; 21790 of May 30, 1885 to Samuel Roebuck; 22335 of Sep. 1, 1885 to L. G. Allen; 24701 of Aug. 10, 1886 to Benjamin Harris and P. W. Dunning; 24797 of Aug. 23, 1886 to E. A. Horton; 25521 of Dec. 7, 1886 to Paraffine Paint Co.; 26668 of May 9, 1887 to Daniel Brobst; 26765 of May 23, 1887 to J. B. Courville, Jr.; 27939 of Nov. 5, 1887 to James Murray; 37601 of Oct. 15, 1891 to J. B. Zook; 38562 of Mar. 22, 1892 to E. W. Grafton; 45534 of Mar. 14, 1894 to Hjolmar Johanson; 45614 of Mar. 22, 1894 to G. H. McAlpine; 60583 of Jul. 12, 1898 to M. A. Benjamin; 62615 of Feb. 9, 1899 to D. F. Lucas; 65194 of Dec. 4, 1899 to C. N. Nielsen; 69284 of Nov. 12, 1900 to W. E. Harris; 84278 of Dec. 8, 1903 to G. E. Reynolds; 85007 of Jan. 26, 1904 to Christian Schallberger; 93634 of Jun. 13, 1905 to Harry de Wallace; 102110 of Nov. 20, 1906 to R. H. Goddin; 114769 of Oct. 27, 1908 to Hydrocarbon Co.; 133091 of May 16, 1911 to Gottlieb Bettschen; 135474 of Sep. 12, 1911 to M. S. Watt and J. D. Campbell; 138138 of Jan. 30, 1912 to H. C. Engelson and Robert Archment; 142502 of Aug. 27, 1912 to J. T. Murphy and Arthur McBrearty; 153308 of Jan. 27, 1914 to T. J. Hadley; 163343 of Jun. 22, 1915 to G. L. Davie and W. L. Windsor-Richards; 182123 of Feb. 5, 1918 to G. R. Strickle; 191684 of Jul. 22, 1919 to E. W. Loftus; 216905 of Mar. 14, 1922 to J. A. Locke and C. S. Hathaway; 218114 of May 2, 1922 to T. J. Duffin; 328242 of Dec. 6, 1932 to W. E. Smith; 331634, 331635 and 331636 of Apr. 11, 1933 to Canadian Industries, Ltd.; 340130 of Mar. 20, 1934 to G. B. Dunford; **Brit. Pats.** of 1868 (Jun. 13), 1932 to Charles Humfrey; of 1887 (Nov. 1), 12632 to J. H. Lyman; of 1897 (Sep. 11), 20902 to William Walters; of 1914 (Mar. 31), 8138 to T. J. Hadley; 195995 of Oct. 11, 1921 to Daniel Gardner; 361040 of Sep. 5, 1930 to S. Asobe; 377484 of Sep. 9, 1931 to R. J. Knight; 378051 of Feb. 6, 1931 to T. F. King; **Ger. Pats.** 93309 of Oct. 18, 1895 to Pierre Janssen; 312064 of Jun. 21, 1918 to Eduard Herzinger and Edmund Naylor; 570486 of Jan. 28, 1931 to Paul Lechler; **French Pat.** 788249 of Oct. 7, 1935 to Société du gaz de Paris.

168 [p. 740] "Water-gas and Coal-gas Tar Paints," by J. Hinds, *Reclamation Rec. of the U. S.*, **12**, 21 (1921).

169 [p. 740] "Bitumen Paints and Red Lead Primers," by Hans Hebberling, *Farben-Ztg.*, **40**, 908 (1935); "Anticorrosion Paints," by E. Assheton, *Paint Manuf.*, **6**, 278 (1936).

170 [p. 740] **U. S. Pats.** 1536550 of May 5, 1925 to J. H. Young; 1790643 of Feb. 3, 1931 to S. U. McGrary, W. F. Rogers and Stanley Gill.

171 [p. 740] **Ger. Pat.** Appl. M–118147 of Dec. 24, 1931 to A. F. Malchow, A.-G.

172 [p. 740] **U. S. Pats.** 659076 of Oct. 2, 1900 to Frederick Lennard; 2060447 of Nov. 10, 1936 to Karl Schoenemann; **Can. Pat.** 218877 of May 23, 1922 to Barrett Co.; **Brit. Pats.** of 1915 (Dec. 6), 17140 to J. L. Mason; 201249 of Apr. 29, 1922 to W. S. Smith; **Ger. Pats.** 98278 of Nov. 25, 1896 to Albert Lessing; 273664 of Sep. 25, 1913 to A. Michel; 399280 of Mar. 29, 1923 to R. J. Lacau; 534644 of Apr. 6, 1928 to Kohlenveredlung und Schwelwerke, A.-G.

173 [p. 740] "Finishing Air-Drying Insulating Varnish for Electrical Purposes," British

Standard Spec. No. 634–1935, British Standards Institution; **Brit. Pat.** of 1907 (Jan. 22), 1678 to J. Connolly.

174 [p. 740] **U. S. Pat.** 2028798 of Jan. 28, 1936 to J. H. Murch; 2062234 of Nov. 24, 1936 to S. C. Robison.

175 [p. 740] "Standard Specifications for the Toxic Ingredients of Anti-Fouling Paints (Dry Cuprous Oxide, Dry Mercuric Oxide)," (D 277–31), A.S.T.M. Standards 1936, II, 598.

176 [p. 741] "Anticorrosion and Anti-fouling Compositions," by P. E. Bowles, *J. Soc. Chem. Ind.*, **41**, 492R (1922); "Report of Sub-Committee XXIII on Anti-Fouling Paints," Proc. A.S.T.M., **22** (1922); "Anti-fouling Paints," by A. M. Muckenfuss, Proc. A.S.T.M., **27**, Part I, 430 (1927).

177 [p. 741] **U. S. Pats.** 190761 of May 15, 1877 to W. P. Jenney; 824069 of Jun. 19, 1906 to F. E. Dunnett; **Can. Pat.** 97963 of Mar. 13, 1906 to F. E. Dunnett; **Ger. Pats.** 192210 of Oct. 14, 1905 to F. E. Dunnett; 517579 of Oct. 17, 1928 to Chemieprodukte, G.m.b.H.

178 [p. 741] **U. S. Pat.** 1762069 of Jun. 3, 1930 to Robert Metzger; **Can. Pat.** 1845 of Nov. 30, 1872 to S. A. Gilman; **Brit. Pat.** 231411 of Apr. 25, 1924 to Paul Lechler.

179 [p. 741] **Can. Pat.** 109900 of Jan. 28, 1908 to J. F. Ouden.

180 [p. 741] **U. S. Pat.** 678201 of Jul. 9, 1901 to Christian Schallberger.

181 [p. 741] "Asphalt Emulsion Paints," by J. M. Fain and F. D. Snell, *Paint, Oil & Chem. Rev.*, **98**, Nos. 13, 14 and 16 (1936); **U. S. Pats.** 788857 of May 2, 1905 to G. A. Thubé and Louis Préaubert; 790821 of May 23, 1905 to H. V. Dunham; 812593 of Feb. 13, 1906 to Louis Préaubert and G. A. Thubé; 1302810 of May 6, 1919 to Lester Kirschbraun; 1469563 of Oct. 2, 1923 to Lester Kirschbraun; 1487471 of Mar. 18, 1924 to Rudolf Plönnis; 1691765 of Nov. 13, 1928 to Lester Kirschbraun; 1733493 of Oct. 29, 1929 to Lester Kirschbraun; 1833612 of Nov. 24, 1931 to H. E. Lloyd; 2010436 of Aug. 6, 1935 to J. A. E. McClave and D. H. Grant; **Can. Pats.** 192491 of Sep. 2, 1919 to Robert Illeman and J. A. Montgomerie; 289642 of May 14, 1929 to Flintkote Co.; 363190 of Jan. 5, 1937 to International Bitumen Emulsions Corp.; **Brit. Pats.** 243976 of Feb. 5, 1925 to Wales Dove Bitumastic, Ltd.; 255684 of Oct. 1, 1925 to W. J. McGivern; 255911 of Jan. 23, 1925 to W. E. Billinghame; 274955 of Apr. 28, 1926 to Colas Products, Ltd.; 319648 of Aug. 22, 1928 to Bowran & Co., Ltd. and J. W. Craggs; 320160 of Jul. 23, 1928 to J. de B. W. Gardiner; 321948 of Jun. 22, 1928 to C. H. Grimshaw; 369242 of Nov. 8, 1930 to W. W. Groves; 381286 of Aug. 18, 1931 to N. V. Tot Voortzetting der Zaken Van Pieter Schoen & Zoon; 445865 of Oct. 15, 1934 to Alfred Tabary; **Ger. Pats.** 312690 of May 10, 1912 to W. Plinatus; 321113 of Jul. 3, 1917 to E. Stephani; 398793 of May 25, 1922 to Ernst Stern; 404356 of Aug. 27, 1922 to Ernst Stern; 405930 of Nov. 22, 1922 to Ernst Stern; 421237 of May 3, 1924 to A. Knecht; 537792 of Aug. 19, 1927 to Eduard Pree; 582608 of Oct. 27, 1929 to C. H. Grimshaw; 582676 of Apr. 17, 1929 to Deutsche Asphalt A.-G. der Limmer und Verwohler Grubenfelder; 598058 of Jun. 18, 1930 to Ferdinand Leiber; Appl. L-85635 of Mar. 31, 1934 to Paul Lechler; 618284 of Aug. 15, 1930 to Twitchell Process Co.; **French Pats.** 683967 of Oct. 26, 1929 to C. H. Grimshaw; 770009 of Sep. 6, 1934 to Les Travaux Souterrains; **Danish Pat.** 33864 of Mar. 14, 1924 to H. Lange.

182 [p. 741] **Ger. Pat.** Appl. G-77791 of Oct. 26, 1929 to C. H. Grimshaw.

183 [p. 741] **U. S. Pats.** 1765676 of Jun. 24, 1930 to L. C. Jones; 2068966 of Jan. 26, 1937 to R. R. Thurston and R. J. Ruble; **Can. Pat.** 313483 of Jul. 21, 1931 to Flintkote Co.; **Brit. Pat.** 360385 of Apr. 28, 1930 to N. V. de Bataafsche Petroleum Maatschappij.

184 [p. 741] **U. S. Pats.** 1487471 of Mar. 18, 1924 to Rudolf Plönnis; 1573896 and 1573897 of Feb. 23, 1926 to W. H. Alton.

185 [p. 741] **U. S. Pat.** 1541130 of Jun. 9, 1925 to Dozier Finley.

186 [p. 741] **U. S. Pat.** 1988921 of Jan. 22, 1935 to L. E. Seng.

187 [p. 741] "Protective Coatings," by P. Rohland, *Farben-Ztg.*, **20**, 1280 (1915); **U. S. Pat.** 1733497 of Oct. 29, 1929 to Lester Kirschbraun; **Can. Pat.** 313275 of Jul. 14, 1931 to Flintkote Co.; **Brit. Pats.** 341443 of Oct. 9, 1929 to N. V. de Bataafsche Petroleum Maatschappij and H. D. Elkington; 369242 of Nov. 8, 1930 to I. G. Farbenindustrie, A.-G.

188 [p. 741] **U. S. Pat.** 1794522 of May 3, 1931 to Lester Kirschbraun; 2068995 of Jan. 26, 1937 to K. E. H. Rodwell; **Can. Pat.** 322473 of May 17, 1932 to Flintkote Corp.

189 [p. 741] **U. S. Pat.** 1893599 of Jan. 10, 1933 to A. W. Seymour.

190 [p. 741] **U. S. Pat.** 1762069 of Jun. 3, 1930 to Robert Mezger; **Ger. Pats.** 398793 of May 25, 1922 to Ernst Stern; 407199 of Apr. 26, 1924 to Paul Lechler.

191 [p. 742] **U. S. Pat.** 1699536 of Jan. 22, 1929 to L. C. Jones, C. C. Loomis and H. W. Banks; **Ger. Pats.** 296271 of Aug. 3, 1916 to Carl Roth; 298708 of Oct. 8, 1916 to Carl Roth.

192 [p. 742] **U. S. Pats.** 1472393 of Oct. 30, 1923 to W. T. Headley; 1793957 of Feb. 24, 1931 to R. K. Painter; 1803488 of May 5, 1931 to E. S. Ross; **Brit. Pats.** 251323 of Jan. 1, 1925 to G. S. Hay; 255684 of Oct. 1, 1925 to W. J. McGivern; 351904 of Dec. 16, 1929 to I. G. Farbenindustrie, A.-G.; 365844 of Feb. 25, 1931 to Erwin Meyer; **Ger. Pats.** 127582 of Jun. 17, 1900 to Carl Baswitz; 170133 of Jun. 3, 1904 to Karl Mann; 336826 of May 29, 1918 to Dr. Plönnis & Co.; 386158 of Apr. 29, 1921 to G. Borle; 407199 of Apr. 26, 1924 to Paul Lechler; 530215 of Feb. 16, 1929 to N. V. Mijnbouw-en Cultuur-maatschappij "Boeton"; 549248 of Mar. 31, 1929 to Uddeholms Aktiebolag Skoghalls-Verken; **French Pat.** 598020 of Jan. 30, 1925 to Asphalt Cold Mix, Ltd.

193 [p. 742] "Acid-proof Coatings for Concrete Surfaces," Letter Circular 42 (Revised), Bureau of Standards, Wash., D. C., (Feb. 12, 1923); **U. S. Pat.** 1382595 of Jun. 21, 1921 to A. C. Biernatzki.

194 [p. 742] **U. S. Pat.** 1954858 of Apr. 17, 1934 to G. H. Alvey.

195 [p. 743] **Brit. Pat.** 396545 of Jul. 7, 1932 to Highway Construction, Ltd.

196 [p. 744] **U. S. Pat.** 1579729 of Apr. 6, 1926 to J. F. Nachod; **Can. Pat.** 254467 of Oct. 6, 1925 to J. H. Russell.

197 [p. 744] **U. S. Pat.** 1382595 of Jun. 21, 1921 to A. C. Biernatzki.

198 [p. 744] **Brit. Pat.** 367782 of Nov. 18, 1930 to I. G. Farbenindustrie, A.-G.

199 [p. 744] **Brit. Pat.** 366559 of Oct. 31, 1930 to L. Lewis.

200 [p. 744] **U. S. Pats.** 145705 of Dec. 16, 1873 to Horace Wheeler; 1509478 of Sep. 23, 1924 to C. E. Kraus.

201 [p. 744] **Ger. Pat.** 98071 of May 31, 1898 to Chemische Fabrik Zimmer & Gottstein.

202 [p. 744] **U. S. Pats.** Reissues 5948A, 5948B and 6191 of Jun. 30, 1874 to H. W. Johns; 76773 of Apr. 14, 1888 to H. W. Johns; 2008667 of Jul. 23, 1935 to F. S. Fleckenstein; **Brit. Pat.** 157217 of Mar. 23, 1922 to Max Rogler.

203 [p. 744] **Brit. Pat.** 153089 of Oct. 29, 1920 to C. F. Curtis.

204 [p. 744] **Brit. Pat.** 125492 of Apr. 12, 1918 to S. G. Kelsey.

205 [p. 745] **Brit. Pat.** 217542 of Jun. 14, 1923 to N. E. Newman.

206 [p. 745] **U. S. Pat.** 1253454 of Jan. 15, 1918 to Herbert Abraham.

207 [p. 745] **U. S. Pat.** 1883683 of Oct. 18, 1932 to E. W. Gardner and D. H. Felder; **Can. Pat.** 333457 of Jun. 20, 1933 to Texaco Development Corp.

208 [p. 745] **Ger. Pats.** 152758 of Jan. 14, 1903 to Carl Roth; Appl. R-76.30 of Jul. 5, 1930 to Ernst Ruwald.

209 [p. 745] **U. S. Pats.** 1074634 of Sep. 30, 1913 to A. C. Horn; 1080632 of Dec. 9, 1913 to A. C. Horn.

210 [p. 745] **Brit. Pat.** 151666 of Apr. 2, 1919 to C. H. Ivinson; **Ger. Pat.** 572196 of Jun. 14, 1929 to Paul Lechler.

211 [p. 745] **U. S. Pat.** 1997924 of Apr. 16, 1935 to E. D. Curtis.

212 [p. 745] **Ger. Pat.** Appl. V–29338 of Mar. 23, 1933 to Grete Versen.

213 [p. 745] "Federal Spec. for Cement; Bituminous, Plastic," Federal Standard Stock Catalogue, Section IV, (Part 5), SS-C-153 (Aug. 1, 1933).

214 [p. 746] **Ger. Pat.** 542440 of Jan. 7, 1932 to Max Tausent and Hans Eggert.

215 [p. 746] **Brit. Pat.** 341914 of Oct. 21, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

216 [p. 746] **U. S. Pats.** 1751690 of Mar. 25, 1930 to C. R. Faben; 2029289 of Feb. 4, 1936 to U. B. Bray, L. B. Beckwith and F. S. Scott; **Brit. Pat.** 312467 of May 4, 1928 to F. W. Gough.

217 [p. 746] **U. S. Pats.** 1813992 of Jul. 14, 1931 to Dozier Finley; 1940645 of Dec. 19, 1933 to R. P. Fletcher, Jr.

218 [p. 747] "Varnish Pitches and Their Investigation," by F. Eichstädt, *Farben-Chem.*, 2, 466 (1931); **U. S. Pat.** 802670 of Oct. 24, 1905 to Maximilian Toch.

219 [p. 748] "Federal Spec. for Varnish; Asphalt," Federal Standard Stock Catalogue, Section IV, (Part 5), TT-V-51, (Apr. 28, 1931).

220 [p. 749] "Spec. for Air-Drying Bituminous Varnish," 3–84, issued by the U. S. Army.

221 [p. 749] "The Use of Paracoumarone Resin in Varnishes," by W. W. King, F. W. Bayard and F. H. Rhodes; *J. Ind. Eng. Chem.*, **12**, 549 (1920); **U. S. Pat.** 1360665 of Nov. 30, 1920 to S. P. Miller; **Brit. Pat.** 149982 of Aug. 10, 1920 to Barrett Co.

222 [p. 749] **U. S. Pat.** 1091621 of Mar. 31, 1914 to L. E. Barringer.

223 [p. 749] **U. S. Pat.** 1149171 of Aug. 10, 1915 to Carl Baeder.

224 [p. 749] "Varnishes and Lacquers for Rubber Footwear," by D. D. Wright, *J. Ind. Eng. Chem.*, **25**, 140 (1933).

225 [p. 750] **Can. Pat.** 338331 of Jan. 2, 1934 to Canadian Industries, Ltd.; **Brit. Pat.** 390628 of Mar. 18, 1932 to Imperial Chemical Industries, Ltd.

226 [p. 750] **U. S. Pat.** 1931110 of Oct. 17, 1933 to W. S. Gocher and A. N. Parrett.

227 [p. 750] **U. S. Pat.** 2002634 of May 28, 1935 to H. S. Holt.

228 [p. 750] **U. S. Pat.** 2041496 of May 19, 1936 to S. D. Shinkle.

229 [p. 750] **U. S. Pats.** 1381112 of Jun. 14, 1921 to W. R. Emig; 1658540 of Feb. 7, 1928 to George Sutherland.

230 [p. 751] "Aluminum Stearate in Paint Technology," by H. Wolff and J. Rabinowitz, *Farbe u. Lacke*, **36**, 428 (1931).

231 [p. 751] "Solvents for Automobile Lacquers," by D. B. Keyes, *J. Ind. Eng. Chem.*, **17**, 558 (1925); "Standard Specifications and Tests for Soluble Nitrocellulose," (D 301–33), A.S.T.M. Standards 1936, II, 797; "Standard Methods of Sampling and Testing Lacquer Solvents and Diluents," (D 268–33), A.S.T.M. Standards 1936, II, 813; "Tentative Methods of Test for Soluble Nitrocellulose Base Solutions," (D 365–35T), A.S.T.M. Tentative Standards, 1936, 590; "Tentative Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels," (D 333–34T), A.S.T.M. Tentative Standards 1936, 596; "Standard Specifications for Acetone," (D 329–33), A.S.T.M. Standards 1936, II, 784; "Standard Specifications for Normal Amyl Acetate," (D 318–36), A.S.T.M. Standards 1936, II, 785; "Standard Specifications for Amyl Alcohol (Synthetic)," (D 319–33), A.S.T.M. Standards 1936, II, 787; "Standard Specifications for Ethyl Acetate (85 to 88 per cent Grade)," (D 302–33), A.S.T.M. Standards 1936, II, 793; "Standard Specifications for Ethyl Lactate (Synthetic)," (D 321–33), A.S.T.M. Standards 1936, II, 792; "Standard Specifications for Normal Butyl Acetate (88 to 92 per cent Grade)," (D 303–33), A.S.T.M. Standards 1936, II, 790; "Standard Specifications for Butanol (Normal Butyl Alcohol)," (D 304–33), A.S.T.M. Standards 1936, II, 789; "Standard Specifications for Butyl Propionate (90 to 93 per cent Grade)" (D 320–33), A.S.T.M. Standards 1936, II, 791; "Standard Specifications for Ethylene Glycol Mono Butyl Ether" (D 330–35), A.S.T.M. Standards 1936, II, 794; "Standard Specifications for Ethylene Glycol Mono Ethyl Ether," (D 331–35), A.S.T.M. Standards 1936, II, 795; "Standard Specifications for Acetate Ester of Ethylene Glycol Mono Ethyl Ether (95 to 96 per cent Grade)," (D 343–35), A.S.T.M. Standards 1936, II, 796.

232 [p. 752] "Standard Specifications and Tests for Tricresyl Phosphate," (D 363–36), A.S.T.M. Standards 1936, II, 809.

233 [p. 752] **Brit. Pat.** 430664 of Dec. 22, 1933 to F. B. Dehn.

234 [p. 752] "Nitrocellulose Technology," by E. C. Worden, D. Van Nostrand Co., New York (1911); "Technology of Cellulose Esters," by E. C. Worden, D. Van Nostrand Co., New York (1921); "Cellulose, Celluloseprodukte und Kautschuksurrogate," by Josef Bersch, 2nd Edition, A. Hartleben's Verlag, Vienna and Leipzig (1922); "Pyroxylin Enamels and Lacquers," by S. P. Wilson, D. Van Nostrand Co., New York (1925); "Cellulose Ester Varnishes," by F. Sproxton, D. Van Nostrand Co., New York (1925); "A Survey of Nitrocellulose Lacquer," by B. K. Brown and F. M. Crawford, The Chemical Catalog Co., Inc., New York (1928); "The Application of Cellulose Lacquers and Enamels," by A. E. Robinson, Scott, Greenwood & Son, London (1929); "The Cellulose Lacquers," by Stanley Smith, Sir Isaac Pitman & Sons, Ltd., London (1930); "Nitrocelluloseesterlacke und Zaponlacke," by Fritz Zimmer, Verlag von S. Hirzel, Leipzig (1931); "Celluloseesterlacke," by Calisto

Bianchi and Adolf Weihe, Verlag von Julius Springs, Berlin (1931); "Cellulose Acetate—Its Manufacture and Applications," by A. G. Lipscomb, Ernst Benn, Ltd., London (1933); "The Manufacture of Nitro Cellulose Lacquers," by R. G. Daniels, Leonard Hill, Ltd., London (1933); "Technology of Cellulose Ethers," by E. C. Worden, Chemical Catalog Co., Inc., New York (1933); "Lösungsmittel und Weichmachungsmittel," by T. H. Durrans and Otto Merz, Verlag von Wilhelm Knapp, Halle (Saale), (1933); "Nitrocellulose Ester Lacquers," by Fritz Zimmer, translated by H. K. Cameron, Chapman & Hall, Ltd., London, and D. Van Nostrand Co., Inc., New York (1934); "The Chemistry of Synthetic Resins," by Carleton Ellis, Reinhold Publishing Corp., New York (1935); "Celluloseverbindungen und ihre besonders wichtigen Verwendungsgebiete dargestellt an Hand der Patent-Weltliteratur," by Otto Faust, Verlag von Julius Springer, Berlin (1935); **U. S. Pats.** 91378 of Jun. 15, 1869 to Daniel Spill; 91393 of Jun. 15, 1869 to E. O. W. Whitehouse; 158188 of Dec. 29, 1874 to Alfred Starr; 234675 of Nov. 23, 1880 to C. M. Jacob; 1263186 of Apr. 16, 1918 to W. G. Abbott; 1330421 of Feb. 10, 1920 to H. A. Babis; 1392040 of Sep. 27, 1921 to H. A. Babis; 1397103 of Nov. 15, 1921 to Einar Langfeldt; 1583703 of May 4, 1926 to S. E. Sheppard and J. J. Schmitt; 1885780 of Nov. 1, 1932 to E. T. Stille; **Brit. Pats.** of 1855 (Apr. 3), 745 to L. Cornides; of 1889 (May 22), 8513 to T. Hughes; of 1907 (Oct. 12), 22528 to H. Tas and J. David; of 1908 (Jun. 22), to A. Payne; of 1910 (Jan. 7), 476 to C. Mijnssen; 156527 of Jan. 5, 1921 to Siemens-Schuckert-Werke, G.m.b.H.; 157119 and 157120 of Jan. 8, 1921 to Siemens-Schuckert-Werke, G.m.b.H.; 251323 of Jan. 1, 1925 to G. S. Hay; 430664 of Jun. 24, 1935 to Deutsche Hydrierwerke A.-G.; **Ger. Pats.** 51554 of Nov. 20, 1888 to T. F. Craddock and John Thom; 102962 of Feb. 9, 1898 to F. H. Stracke; 227208 of Jun. 20, 1909 to Wilhelm Winter and Ernst Winkler; 426262 of Apr. 8, 1923 to Cella-Drahtwerke, G.m.b.H.; **French Pat.** 709429 of Mar. 8, 1930 to Le Joint Français.

235 [p. 752] **U. S. Pats.** 967337 of Aug. 16, 1910 to D. T. Day; 1305790 of Jun. 3, 1919 to C. N. Forrest and J. V. Meigs; 1945869 of Feb. 6, 1934 to S. W. Sparks.

236 [p. 752] **Brit. Pats.** of 1911 (Mar. 1), 5165 to British Thomson-Houston Co., Ltd.; of 1911 (Apr. 19), 9559 to J. W. Aylsworth.

237 [p. 752] **Brit. Pat.** of 1911 (Aug. 22), 18818 to C. F. Boehringer & Söhne.

238 [p. 752] **U. S. Pats.** 234675 of Nov. 23, 1880 to C. M. Jacob; 294557 of Mar. 4, 1884 to W. V. Wilson; **Ger. Pat.** 51554 of Jul. 7, 1890 to J. F. Craddock and John Thom.

239 [p. 752] **U. S. Pats.** 239423, 239424 and 239425 of Mar. 29, 1881 to L. S. Beals.

240 [p. 753] **Brit. Pat.** 335247 of Jun. 20, 1929 to J. Y. Johnson.

241 [p. 753] "Use of Tar Varnishes," by A. Pignot, *J. Usines Gaz.*, **56**, 265 (1932).

242 [p. 753] **Austrian Pat.** 100688 of Mar. 15, 1925 to Siemens-Schuchert-Werke, G.m.b.H.; **Swiss Pat.** 97643 of Feb. 16, 1921 to Siemens-Schuckert-Werke, G.m.b.H.

243 [p. 753] **Ger. Pat.** 326411 of Jan. 29, 1916 to Franz Goertz.

244 [p. 753] **Brit. Pats.** 251323 of Jan. 1, 1925 to G. S. Hay; 274955 of Apr. 28, 1926 to Colas Products, Ltd.

245 [p. 754] **U. S. Pats.** 789600 of May 9, 1905 to L. S. Flatau; 1948906 of Feb. 27, 1934 to G. B. Dunford; 2069314 of Feb. 2, 1937 to J. K. Hunt and J. H. Shipp.

246 [p. 754] **U. S. Pat.** 1627069 of May 3, 1927 to V. M. Wade, A.C. Zimmerman and S. D. Heron.

247 [p. 754] **U. S. Pat.** 1906154 of Apr. 25, 1933 to Bennard Hemann.

248 [p. 754] **U. S. Pat.** 1930393 of Oct. 10, 1933 to Jacques Lemerle.

249 [p. 754] **U. S. Pats.** 1934709 of Nov. 7, 1933 to A. M. Alvarado and A. N. Parrett; 1955355 of Apr. 17, 1934 to A. M. Alvarado and A. N. Parrett; 1982231 of Nov. 27, 1934 to A. N. Parrett; 1990474 and 1990475 of Feb. 12, 1935 to A. M. Alvarado; 2047797 of Jul. 14, 1936 to A. N. Parrett; 2047889 of Jul. 14, 1936 to S. J. Roskosky; 2065881 of Dec. 29, 1936 to A. M. Alvarado and H. J. Barrett.

250 [p. 754] "A Handbook of Japanning and Enameling," by W. N. Brown, Scott, Greenwood & Son, London (1901); 2nd Edition (1913); "Motor and Carriage Painting," by A. Bates and C. E. Oliver, The Trades Paper Publishing Co., Ltd., London (1915); "The Application of Gas to Japanning and Lacquering," by E. F. Davis, National Commercial Gas Association, Brooklyn, N. Y. (1916); "Automobile Painting and Carriage and Wagon Paint-

ing," by F. N. Vanderwalker, Frederick J. Drake & Co., Chicago (1919); "Practical Japanning and Enameling," by W. J. Miskella, Finishing Research Laboratories, Inc., Chicago (1928); "Practical Automotive Lacquering," by W. J. Miskella, Finishing Research Laboratories, Inc., Chicago (1928); "Baking Insulating Varnish (Bitumen Type) for Electrical Purposes," British Standard Spec. No. 514-1933, British Standards Institution.

251 [p. 755] "Insulating Varnishes in Electrotechnics," by A. R. Matthis, Preston, Attwater & Sons, London (1929).

252 [p. 755] **U. S. Pat.** 1786824 of Dec. 30, 1930 to P. B. Cochran.

253 [p. 755] **U. S. Pat.** 1091621 of Mar. 31, 1914 to L. E. Barringer.

254 [p. 755] **U. S. Pat.** 1558082 of Oct. 20, 1925 to Daniel Gardner.

255 [p. 755] "Wrinkle Finishes," by G. G. Sward, Circular No. 518, Scientific Section, National Paint, Varnish and Lacquer Assoc., Inc., Wash., D. C. (Sep. 1936).

256 [p. 756] **U. S. Pats.** 1266335 of May 14, 1918 to Buel Smith; 1294422 of Feb. 18, 1919 to W. P. Davey; **Brit. Pat.** 155398 of Sep. 30, 1919 to E. Schou.

257 [p. 756] **U. S. Pats.** 1294627 of Feb. 18, 1919 to W. P. Davey; 1472716 of Oct. 30, 1923 to W. P. Davey; 1723844 of Aug. 6, 1929 to W. P. Davey; 1726473 of Aug. 27, 1929 to W. P. Davey.

258 [p. 756] **Brit. Pat.** 221334 of Aug. 16, 1923 to British Thomson-Houston Co., Ltd.

259 [p. 756] **Can. Pat.** 260231 of Apr. 27, 1926 to Canadian General Electric Co., Ltd.

CHAPTER XXXI

1 [p. 758] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, 948; American Petroleum Institute Standard A.P.I. No: 528–33; American Standards Association Standard A.S.A. No: Z 11.33–1935.

2 [p. 762] "Methods of Analysis Used in the Coal-tar Industry," by J. M. Weiss, *J. Ind. Eng. Chem.*, **10**, 732, 817, 911 and 1006 (1918); Bull. No. 691, U. S. Dept. Agri., Wash., D. C. (Jul. 10, 1918); Bull. No. 940, pp. 73–74, U. S. Dept. Agri., Wash., D. C. (Oct. 10, 1921); "Standard Methods of Sampling Bituminous Materials" (D 140–25), A.S.T.M. Standards 1936, II, 1088; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T–40; "Sampling of Coal Tar and its Products," British Standard Spec. No. 616–1935, British Standards Institution; London; "Methods of Sampling and Examination of Bituminous Road Mixtures," British Standard Spec. No. 598–1935, British Standards Institution, London.

3 [p. 763] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Bottle or Beaker Sampling," 957.

4 [p. 765] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Continuous Sampling," 959; "Standard Methods of Sampling and Testing Creosote" (D38–33), A.S.T.M. Standards 1936, II, 531.

5 [p. 767] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Dipper Sampling," 960.

6 [p. 769] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Thief Sampling," 961.

7 [p. 771] "Standard Method of Sampling Coal" (D 21–16), A.S.T.M. Standards 1936, II, 382; American Tentative Standard A.S.A. No: XI–1921, American Standards Association; "Standard Methods of Laboratory Sampling and Analysis of Coal and Coke" (D 271–33), A.S.T.M. Standards 1936, II, 387; American Tentative Standard A.S.A. No.: K 18–1933, American Standards Association.

8 [p. 771] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Grab Sampling," 968.

9 [p. 772] "Standard Methods of Sampling Petroleum and Petroleum Products" (D 270–33), A.S.T.M. Standards 1936, II, "Borings Sampling," 967.

10 [p. 773] "Tentative Recommended Practice for Bituminous Paving Plant Inspection" (D 290–28T), A.S.T.M. Tentative Standards 1936, 756; "Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Material (Including Some

Material Survey Methods)" (D 75–22), A.S.T.M. Standards 1936, II, 1092; American Standard A.S.A. No.: A 26–1930, American Standards Association.

11 [p. 773] "Tentative Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc, or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules" (D 228–33T), A.S.T.M. Tentative Standards 1936, 852.

CHAPTER XXXII

1 [p. 776] "Methods of Analysis in the Coal-tar Industry," by J. M. Weiss, *J. Ind. Eng. Chem.*, **10**, 732, 817 and 1006 (1918).

2 [p. 776] " Identification of Bituminous Materials," by H. B. Pullar, *Oildom*, **6**, 130 (1916).

3 [p. 780] "Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −20 to +150° C., 0 to +300° F." (D 182–25), A.S.T.M. Standards 1936, II, 1403; "Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300° C., +20 to +580° F." (D 183–25), A.S.T.M. Standards 1936, II, 1405; "Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +400° C., +20 to +760° F." (D 184–25), A.S.T.M. Standards 1936, II, 1407; "Federal Spec. for Thermometers; Industrial," Federal Standard Stock Catalogue, Section IV, (Part 5), GG-T-321, Mar. 31, 1931.

4 [p. 780] "Fluorescence Analysis in Ultra-Violet Light," by J. A. Radley and J. Grant, London (1935).

5 [p. 780] "Asphalt, Bitumen and Tar; Their Respective Uses in Road Construction," by D. M. Wilson, *Chem. & Ind.*, **53**, 924 (1934); "Color Testing of Bitumen," by D. M. Wilson, *Chem. & Ind.*, **54**, 1040 (1935).

6 [p. 780] "Ueber die Färbung der Bitumina, im Besonderen der Asphalte," by Edmund Graefe, *Bitumen*, **4**, 219 (1934); *Petroleum Z.*, **31**, 1 (1935).

7 [p. 781] "Distinguishing Between Natural and Artificial Asphalts," by R. Bratter, *Z. angew. Chem.*, **29**, II, 47 (1916); "Behavior of Asphalt and Tar in Ultra-violet Light," by Walter Becker, *Asphalt u. Teer Strassenbautechnik*, **30**, 87 (1930); "Analysis of Fluorescence in Coal-tar and Petroleum Pitch," by W. Teuscher, *Chem.-Ztg.*, **54**, 987 (1930); "Ueber die Inhomogenitätserscheinungen bei Mischungen von Teer mit Erdölasphalt," by Franz Macht, *Asphalt u. Teer Strassenbautechnik*, **31**, 352 (1931); "Die Lumineszenzanalyse in der Teerindustrie," by W. Kärsten, *Teer u. Bitumen*, **30**, 417 (1932); "Behavior of Bitumens in Filtered Ultra-violet Light," by S. Bruckner and P. Meinhard, *Petroleum*, **29**, 8 (1933); "The Determination of the Proportion of Bitumen and Tar in Mixtures of the Two," by A. B. C. Licence, *J. Soc. Chem. Ind.*, **55**, 487 (1936).

8 [p. 782] "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," by G. L. Oliensis, Proc. A.S.T.M., **33**, Part II, p. 715 (1933); "A Further Study of the Hetrogeneity of Asphalt—A Quantitative Method," by G. L. Oliensis, Proc. A.S.T.M., **36**, Part II, p. 494 (1936).

9 [p. 787] "Diffusion of Water Through Organic Insulating Materials," by D. B. Herrmann, *Rubber Age*, **36**, 73 (1934); *Bell Lab. Record*, **13**, 45 (1934); "Diffusion of Water Through Insulating Materials," by R. L. Taylor, D. B. Herrmann and A. R. Kemp, *J. Ind. Eng. Chem.*, **28**, 1255 (1936).

10 [p. 788] "Standard Definitions of Terms Relating to Specific Gravity" (E 12–27), A.S.T.M. Standards 1936, II, 1436.

11 [p. 790] "Standard Methods for Testing Tar and its Products," Serial No. G.P.1, pp. 19–27, Standardization of Tar Products Tests Committee, Printed by W. Heffer & Sons, Ltd., Cambridge, England (Dec., 1929).

12 [p. 792] "Specific Gravity of Water at Varying Temperatures," Circular No. 19, p. 43, Bureau of Standards, Wash., D. C. (Mar. 30, 1916); "Standard Abridged Volume Correction Table for Petroleum Oils" (D 206–36), A.S.T.M. Standards 1936, II, 988; American Standard A.S.A. No: Z 11.1–1936, American Standards Association; American Petroleum Institute Standard A.P.I. No: 500–36.

13 [p. 793] "Standard Method of Test for Specific Gravity of Creosote Oil" (D 368–33), A.S.T.M. Standards 1936, II, 548; American Assoc. State Highway Officials' Standard, A.A.S.H.O. No: T-82; "Standard Method of Test for Specific Gravity, 38/15.5 C., of Creosote Fractions" (D 369–33), A.S.T.M. Standards 1936, II, 550; American Assoc. State Highway Officials' Standard, A.A.S.H.O. No: T-74; "Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer" (D 287–36), A.S.T.M. Standards 1936, II, 918; American Standard A.S.A. No: Z 11.31–1936, American Standards Association; American Petroleum Standard A.P.I. No: 526–36.

14 [p. 794] "Specific Gravity—Its Determination for Tars, Oils and Pitches," by J. M. Weiss, *J. Ind. Eng. Chem.*, 7, 21 (1915); "Methods for the Examination of Bituminous Road Materials," by Prévost Hubbard and C. S. Reeve; Bull. No. 314, U. S. Dept. Agri., Wash., D. C. (Dec. 10, 1915); "Laboratory Manual of Bituminous Materials," by Prévost Hubbard, p. 30, John Wiley & Sons, Inc., New York (1916); "Bestimmung des specifischen Gewichtes von Starren Körpern," by W. Maass, *Petroleum*, 27, Supp., 3 (1931).

15 [p. 794] "Determination of the Specific Gravity of Asphalt by Means of a Hydrometer," by H. R. Berry, E. H. Smith and F. R. Lang, *J. Ind. Eng. Chem.*, Analytical Edit., 1, 164 (1929).

16 [p. 794] "Standard Density and Volumetric Tables," Circ. No. 19, Bureau of Standards, Wash., D. C. (1916); "U. S. Standard Baumé Hydrometer Scales," Circ. No. 59, Bureau of Standards, Wash., D. C. (1916).

17 [p. 795] "Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer" (D 287–36), A.S.T.M. Standards 1936, II, 918; American Standard A.S.A. No: Z 11.31–1936, American Standards Association; American Petroleum Institute Standard A.P.I. No: 526–36; "National Standard Petroleum Oil Tables," Table V, Circ. No. C–410, Bureau of Standards, Wash., D. C. (1934); "Abridged Volume Correction Table for Petroleum Oils," Revised Suppl. Circ. No. 154, Bureau of Standards, Wash., D. C. (1930).

18 [p. 796] "Specific Gravity—Its Determination for Tars, Oils and Pitches," by J M. Weiss, *J. Ind. Eng. Chem.*, 7, 21 (1915).

19 [p. 796] "Method for Determining the Density of Petroleum Residuums," by Cr. Predescu, *Bull. Soc. Acad. Roumaine*, 6, 148 (1920).

20 [p. 796] "Methods for Testing Coal Tars and Refined Tars, Oils and Pitches Derived Therefrom," by S. R. Church, *J. Ind. Eng. Chem.*, 3, 228 (1911); 5, 195 (1913); "Method for the Examination of Bituminous Road Materials," by Prévost Hubbard and C. S. Reeve, Bull. No. 314, U. S. Dept. Agri., p. 5, Wash., D. C. (Dec. 10, 1915); "Laboratory Manual of Bituminous Materials," by Prévost Hubbard, p. 34, John Wiley & Sons, Inc., New York (1916).

21 [p. 796] "Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches" (D 70–27), A.S.T.M. Standards 1936, II, 1110.

22 [p. 798] "Methods of Analyzing Coal and Coke," Tech. Paper No. 8, Bureau of Mines, Wash., D. C. p. 37 (1913).

23 [p. 799] "Standard Method of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments" (D 71–27), A.S.T.M. Standards 1936, II, 1108.

24 [p. 802] See Chapter III, Reference 5.

25 [p. 805] "Kolloidchemie," by Richard Zsigmondy, 5th Edition, Verlag von Otto Spamer, Leipzig (1925); "Ultramicroscopic Examination of Asphalts," by F. J. Nellensteyn and J. P. Kuipers, *Chem. Zentralblatt*, I, 1771 (1929); *Kolloid-Zeitschr.*, 47, 155 (1929); "Handbook of Chemical Microscopy," by E. M. Chamot and C. W. Mason, (Vols. I & II), John Wiley & Sons, Inc., New York (1930); "Colloid Chemistry," by Jerome Alexander, Vol. III, The Chemical Catalog Co., Inc., New York (1931).

26 [p. 805] "Ultra-microscopic Examination of Disperse Colloids Present in Bituminous Road Materials," by E. C. E. Lord, *J. Agri. Research*, 17, 167 (1919).

27 [p. 809] "Microscopic Researches on Coal-tar Solutions," by F. J. Nellensteyn, *Z. Angew. Chem.*, 43, 402 (1930); *Gas World*, 92, 54 (1930).

28 [p. 809] "Die Mikronenzahl," by F. J. Nellensteyn and J. P. Kuipers, *Teer u. Bitumen*, 31, 309 (1933).

29 [p. 810] "An Investigation into the Nature and Properties of Coal Tar," by E. V. Evans and H. Pickard, published by South Metropolitan Gas Co., London (1931); "The Measurement of the Viscosity of Coal Tars and Pitches," by H. Pickard, published by South Metropolitan Gas Co., London (1932).

30 [p. 810] "The Temperature-Time-Pressure Law," by W. Eymann, *Asphalt Teer Strassenbautech.*, **33**, 751 (1933); "The Viscosity of Road Tars," by J. G. Mitchell and A. R. Lee, *J. Soc. Chem. Ind.*, **54**, 407T (1935); **55**, 167T (1936); "Die Qualifizierung der im Strassenbau gebrauchten Bitumina auf Grund ihre absoluten Viskosität," by József Cságoly, *Asphalt u. Teer Strassenbautechnik*, **35**, 667 and 687 (1935); "Physical Properties of Coal Tars," by E. W. Volkmann and E. O. Rhodes, *J. Ind. Eng. Chem.*, **28**, 721 (1936).

31 [p. 810] "Determination of Viscosity in Absolute Units," British Standard Spec. No. 188–1929 (Add. Sep. 1929), British Standards Institutition; "The Viscosity of Tar—Its Significance in the Surfacing of Roads," by G. H. Fuidge, *J. Soc. Chem. Ind.*, **55**, 301 (1936).

32 [p. 810] "Standard Methods for Testing Tar and its Products," published by the Standardizazion of Tar Products Tests Committee, p. 63, London (Dec. 1929).

33 [p. 811] "Standard Specifications for Thermometers for Engler Viscosimeters" (D 300–34), A.S.T.M. Standards 1936, II, 1409.

34 [p. 812] "Untersuchung der Kohlenwasserstofföle und Fette," by David Holde, p. 136, Verlag von Julius Springer, Berlin (1913); "Methods for the Examination of Bituminous Road Materials," by Prévost Hubbard and C. S. Reeve, Bull. No. 314, p. 7, U. S. Dept. Agri., Wash., D. C. (Dec. 10, 1915).

35 [p. 812] "Standard Methods of Test for Viscosity by Means of the Saybolt Viscosimeter" (D 88–36), A.S.T.M. Standards 1936, II, 982; American Standard A.S.A. No: Z 11.2–1936, American Standards Association; Petroleum Institute Standard A.P.I. No: 518–36.

36 [p. 817] "Fluidity and Plasticity," by E. C. Bingham, McGraw-Hill Book Co., New York (1922); "A New Combined Viscosimeter and Plastometer," by E. C. Bingham and H. A. Murray, Jr., Proc. A.S.T.M., **23**, Part II, 655 (1923); "A Modified Plastometer for Industrial Use," by D. V. Gregory, *J. Rheology*, **1**, 30 (1929); "New Viscosimeter for Bitumens Has Extended Range," by E. O. Rhodes, E. W. Volkmann and C. T. Barker, *Eng. News-Record*, **115**, 714 (1935); "Measurement of High Viscosity—A Rapid Method," by R. N. Traxler and H. E. Schweyer, Proc. A.S.T.M., **36**, Part II, 518 (1936); **U. S. Pat.** 2076591 of Apr. 13, 1937 to E. O. Rhodes, E. W. Volkmann and C. T. Barker.

37 [p. 819] "A New English Tar Tester," by Anon., *Good Roads*, **3**, 337 (1912); **Brit. Pat.** of 1911 (Oct. 6), 22042 to John Hutchinson.

38 [p. 820] "The Variation of the Hutchinson Consistency of Tars with Temperature," by H. M. Spiers, *J. Soc. Chem. Ind.*, **45**, 396T (1926).

39 [p. 820] "The Consistency of Bituminous Mixtures," by H. M. Spiers, *J. Soc. Chem. Ind.*, **45**, 399T (1926).

40 [p. 820] "Standard Method of Float Test for Bituminous Materials" (D 139–27), A.S.T.M. Standards 1936, II, 1061; American Standard A.S.A. No: A 37.2–1930, American Standards Association.

41 [p. 823] "Some Relations Between the Characteristics of Straight Distilled Tar Residues," by J. W. Kennedy, Proc. A.S.T.M., **23**, Part II, 341 (1923).

42 [p. 823] "Methods for Testing Coal Tar and Refined Tars, Oils and Pitches Derived Therefrom," by S. R. Church, *J. Ind. Eng. Chem.*, **3**, 229 (1911).

43 [p. 823] "Determination of the Viscosity of Tar, Pitch, and Similar Opaque Substances," by D. C. Broome and A. R. Thomas, *J. Soc. Chem. Ind.*, **50**, 424T (1931); "An Automtaic Modified Falling-Sphere Viscosimeter," by B. A. Jones, *J. Ind. Eng. Chem.*, Anal. Ed., **6**, 80 (1934).

44 [p. 823] "Specification for the Determination of Viscosity in Absolute Units," B. E. S. A., No. 188 (1929).

45 [p. 825] "The Alternating Stress Method for the Measurement of the Consistency of Very Stiff Materials," by E. C. Bingham and R. A. Stephens, *Physics*, **5**, 217 (1934); "Rheological Properties of Asphalts: I. Effect of Temperature," by C. U. Pittman and R. N. Traxler, *Physics*, **5**, 221 (1934); "Rheological Properties of Asphalts: II. Discussion of Penetra-

tion-Viscosity Relationships," by R. N. Traxler, C. U. Pittman and F. B. Burns, *Physics*, 6, 58 (1935); "Rheological Properties of Asphalts: III. A Viscosity Index," by R. N. Traxler and H. E. Schweyer, *Physics*, 7, 67 (1936); "Measurement of High Viscosity—A Rapid Method," by R. N. Traxler and H. E. Schweyer, Proc. A.S.T.M., 36, Part II, 518 (1936); "Viscosities of Liquid-Solid Systems," by R. N. Traxler, H. E. Schweyer and L. R. Moffatt, *J. Ind. Eng. Chem.*, 29, 489 (1937).

46 [p. 826] "A Method for Determining the Internal Friction of Hard Substances," by M. Segel, *Phys. Zeit.*, 4, 493 (1903); "Viscosity and Elastictiy of Highly Viscous Materials," by A. Pochettino, *Nuovo cimento*, 8, 77 (1914); "Colloid Chemistry of Asphalts," by C. J. Mack; *J. Phys. Chem.*, 36, 2901 (1932); "Measurement of High Viscosity—A Rapid Method," by R. N. Traxler and H. E. Schweyer, Proc. A.S.T.M., 36, Part II, 518 (1936).

47 [p. 826] "Studies Concerning the Friction of Liquids," by M. Couette, *Ann. chimie et de physique*, 21, No. 6, 433 (1890); "Investigations into the Plastic Properties of Asphaltic Bitumen," by R. N. J. Saal and G. Koens, *J. Inst. Petrol. Technologists*, 19, 176 (1933); "The Conicylindrical Viscosimeter," by M. Mooney and R. H. Ewart, *Physics*, 5, 350 (1934); "Measurement of High Viscosity—A Rapid Method," by R. N. Traxler and H. E. Schweyer, Proc. A.S.T.M., 36, Part II, 518 (1936).

48 [p. 828] "Tentative Definitions of the Terms Consistency and Plasticity" (E 24–34T), A.S.T.M. Tentative Standards 1936, 1328.

49 [p. 830] "An Apparatus for Determining the Relative Degree of Cohesion of a Semi-Liquid Body," by H. C. Bowen, *School of Mines Quarterly*, 10, 297 (1889); "Report of the Operations of the Engineer Department of the District of Columbia for 1889–1890," p. 106, Wash., D. C. (1890); "Recent Experience with Asphalt in Washington, D. C.," by Anon., *Eng. Record*, 24, 348 (1891); U. S. Pat. 494974 of Apr. 4, 1893 to H. C. Bowen.

50 [p. 830] "Report of the Engineer Department of the District of Columbia for the Year Ending June 30, 1898, p. 127, Wash., D. C. (1898); "Report of the Inspector of Asphalt and Cement of the District of Columbia for the Year Ending June 30, 1901," by A. W. Dow, p. 158, Wash., D. C. (1901); "Testing of Bitumens for Paving Purposes," by A. W. Dow, Proc. A.S.T.M., 3, 354 (1903); "Relation Between Some Physical Properties of Bitumens and Oils," by A. W. Dow, Proc. A.S.T.M., 6, 497 (1906).

51 [p. 830] "The Development of the Penetrometer as Used in the Determination of the Consistency of Semi-solid Bitumens," by Clifford Richardson and C. N. Forrest, Proc. A.S.T.M., 7, 626 (1907); "A Further Development of the Penetrometer as Used in the Determination of the Consistency of Semi-solid Bitumens," by C. N. Forrest, Proc. A.S.T.M., 9, 600 (1909); "Examination of Asphalt," by M. Gary, *Mitt. Königl. Materialsprüf.*, 33, 216 (1916).

52 [p. 830] "A New, Electrically Controlled and Timed Asphalt Penetrometer," by H. W. Mahr, *J. Ind. Eng. Chem.*, 6, 133 (1914); "Apparatus for Comparing the Hardness of Pitches and Bitumens," by B. J. Hall and T. G. Marriott, *The Engineer* (Mar. 24, 1922); U. S. Pats. 512687 of Jan. 16, 1894 to A. W. Dow and T. R. Griffith; 1225438 of May 8, 1917 to W. B. Howard.

53 [p. 831] "A New Automatic Penetrometer," by Anon., *Roads and Road Construction*, 10, No. 118, 310 (1932).

54 [p. 833] "Effect of the Diameter of Bitumen Holder on the Penetration Test," by C. S. Reeve, Proc. Int. Assoc. Testing Materials, 6th Congress, XXV-3, N. Y. (1912).

55 [p. 833] "Report on Penetration of Bituminous Materials," Proc. A.S.T.M., 15, Part I, 352 (1915); 16, Part I, 306 (1916); "Revised Standard Tests for Penetration of Bituminous Materials," by L. W. Page, *Chem. Eng. Manuf.*, 24, 32 (1916).

56 [p. 833] "A New Penetration Needle for Use in Testing Bituminous Materials," by C. S. Reeve and F. P. Pritchard, *J. Agri., Research*, 5, 1121 (1916); "Asphalt Penetrometer," by A. Wolf, *J. Sci. Instr.*, 9, 22 (1932).

57 [p. 833] "Effect of Controllable Variables on the Penetration Test for Asphalts and Asphalt Cements," by Prévost Hubbard and F. P. Pritchard, *J. Agri. Research*, 5, 805 (1916).

58 [p. 833] "Determination of the Penetration of Asphalts," by Edmund Graefe, *Petroleum*, 24, 1599 (1928); "Standard Method of Test for Penetration of Bituminous Materials"

(D 5–25), A.S.T.M. Standards 1936, II, 1080; American Standard A.S.A. No: A 37.1–1930 American Standards Association.

59 [p. 835] "Change of Penetration with Temperature of Various Asphalts," by I. Bencowitz and E. S. Boe, *J. Ind. Eng. Chem.* (Analytical Edition), **8**, 157 (1936).

60 [p. 836] "Tentative Method of Test for Penetration of Greases and Petrolatum" (D 217–33T), A.S.T.M. Tentative Standards 1936, 637; American Tentative Standard A.S.A. No: 11.3–1933 American Standards Association.

61 [p. 840] "A New Consistometer and its Application to Greases and to Oils at Low Temperatures," by Ronald Bulkley and F. G. Bitner, Research Paper No. 188, *Bureau of Standards J. of Research*, **5**, (Jul. 1930).

62 [p. 840] "A Micropenetrometer: Its Application to the Testing of Fats," by Egbert Freyer, *J. Ind. Eng. Chem.*, (Analytical Ed.), **2**, 423 (1930).

63 [p. 840] "An Apparatus and Method for Measuring the Consistency of Roofing Putties and Fibrous Roof Coatings," by G. W. Clarvoe, Proc. A.S.T.M., **32**, Part II, 689 (1932).

64 [p. 840] "Bituminous Plastics: Determination of Flow Properties," by R. N. Taxler, *J. Ind. Eng. Chem.* (Analytical Ed.), **8**, 185 (1936).

65 [p. 844] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, Proc. A.S.T.M., **9**, 568 (1909); **11**, 676 (1911); **U. S. Pat.** 989471 of Apr. 11, 1911 to Herbert Abraham.

66 [p. 845] "Plastic Properties of Asphaltic Bitumens," by R. N. J. Saal, *Chem. Weekblad*, **32**, 435 (1935); "Relation between Absolute Viscosity and Penetration of Asphaltic Bitumens," by R. N. J. Saal and J.W. A. Labout, *Physics*, **7**, 408 (1936).

67 [p. 845] "The Viscosity Temperature Susceptibility Coefficient as an Index of the Effect of Temperature on the Consistency of Asphalts," by H. G. Nevitt and L. C. Krchma, presented at the Petroleum Division, Am. Chem. Soc., Kansas City, Mo., Apr. 13–17, 1936; "The Effect of Temperature on the Consistency of Asphalts," by H. G. Nevitt and L. C. Krchma, *J. Ind. Eng. Chem.* (Analyt. Edition), **9**, 119 (1937); "Physical Properties of Asphalt (Viscosity-Temperature Susceptibility)," by Seward Mason, R. J. Loomis and S. D. Patterson, also H. G. Nevitt and L. C. Krchma, *J. Ind. Eng. Chem.* (Analyt. Edition), **9**, 138 (1937).

68 [p. 845] "The Susceptibility of Asphalts to Temperature Changes, by H. E. Schweyer, C. E. Coombs and R. N. Traxler, Proc. A.S.T.M., **36**, Part II, 531 (1936)

69 [p. 847] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, Proc. A.S.T.M., **11**, 683 (1911).

70 [p. 847] "Measuring the Susceptibility of Asphalts to Temperature Changes," by A. Holmes, J. O. Collins and W. C. Child, *J. Ind. Eng. Chem.* (Analyt. Ed.), **8**, 100 (1936); "Change of Penetration with Temperature of Various Asphalts," by I. Bencowitz and E. S. Boe, *J. Ind. Eng. Chem.* (Analyt. Ed.), **8**, 157 (1936); "Rheological Properties of Asphaltic Bitumens," by J. P. Pfeiffer and P. M. van Doormaal, *J. Inst. Petroleum Tech.*, **22**, 414 (1936).

71 [p. 848] "The Testing of Bitumens for Paving Purposes," by A. W. Dow, Proc. A.S.T.M., **3**, 352 (1903); "Report of the Commissioners of the District of Columbia for the Year Ending June 30, 1904," p. 42 (1904); "Methods for Testing Asphalt," by A. W. Dow, *Chem. Eng.*, **1**, 330 (1905); "Tests of Asphalts for Paving Purposes," by A. W. Dow and F. P. Smith, *Munic. Eng.*, **40**, 437 (1911); "Note on Ductility Test of Asphalt," by T. L. Crossby, *J. Ind. Eng. Chem.*, **8**, 89 (1916); "Examination of Asphalt," by M. Gary, *Mitt. Königl. Materialsprüf.*, **33**, 210 (1916).

72 [p. 848] "Standard Method of Test for Ductility of Bituminous Materials" (D 113–35), A.S.T.M. Standards 1936, II, 1058; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-51.

73 [p. 849] "Amalgamating Metal Used in Molding Asphalts," by L. J. Catlin, *J. Ind. Eng. Chem.*, **17**, 521 (1925).

74 [p. 849] "Substitute for Amalgamation in Testing Bituminous Materials for Melting Point, Ductility and Float Test," by Hans Eisner, *J. Ind. Eng. Chem.*, Analytical Ed., **2**, 176 (1930).

75 [p. 850] "Note on Ductility Test of Asphalt," by T. L. Crossley, *J. Ind. Eng. Chem.*, **8**, 89 (1916).

76 [p. 851] "Steinkohlenteerweichpech-Asphaltbitumen," by H. Walther, *Teer u. Bitumen*, **32**, 384 (1933).

77 [p. 851] "Machine for Testing the Ductility of Bituminous Paving Cements," by F. P. Smith, Proc. A.S.T.M., **9**, 594 (1909).

78 [p. 852] "The Cementing Value of Bituminous Binders," by Lester Kirschbraun, *J. Ind. Eng. Chem.*, **6**, 976 (1914); **U. S. Pat.** 1180506 of Apr. 25, 1916 to Lester Kirschbrun.

79 [p. 852] "Low-temperature Ductility of Filler-grade Asphalts," by W. F. Smith. Proc. A.S.T.M., **27**, Part II, 480 (1927).

80 [p. 852] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, Proc. A.S.T.M., **10**, 444 (1910); **11**, 679 (1911).

81 [p. 855] "Adhesion in Relation to Bituminous Road Materials," by A. R. Lee. *J. Soc. Chem. Ind.*, **55**, 23T (1936).

82 [p. 856] "A New Machine for Testing Pitch," by T. Y. Olsen, Proc. A.S.T.M., 10, 592 (1910); "Impact Testing Machine for Pitch," by W. H. Fulweiler, Am. Assoc. Adv. Sci., Wash., D. C. (1911); *Good Roads*. 3, 81 (1912).

83 [p. 856] "Private communication from Clarence B. Osborne, California Highway Commission, Sacramento, Cal.; *Eng. Rec.*, **71**, 329 (1915).

84 [p. 856] "Present Status of Adhesive and Cohesive Tests of Bituminous Materials," by J. S. Crandall, *Science*, **41**, 801 (1915).

85 [p. 856] "Der Paraffingehalt des Bitumens," by Paul Riehm, *Teer u. Bitumen*, **29**, 530 (1931).

86 [p. 856] "Tests of Bituminous Compounds for Electrical Apparatus: Adhesiveness and Plastic Flow," by E. B. Wedmore, Proc. World Petroleum Cong., **2**, 593 (1934).

87 [p. 857] "Ueber die Temperaturabhängigkeit der Molekularen freien Oberflächenenergie von Flüssigkeiten in Temperaturbereich von −80 bis + 1650° C.," by F. M. Jaeger, *Verslag Akad. Wetenschappen*, **23**, 330 (1914); *Z. für anorg. u. allgem. Chemie*, **101**, 18 (1917); "The Surface Tension and the Wetting Ability of Asphalt," by F. J. Nellensteyn, *Chem. Weekblad*, **24**, 54 (1927); "Die Oberflächenspannung-Temperatur-Kurven der Asphaltbitumina und verwandter Produkte," by F. J. Nellensteyn and N. M. Roodenburg, *Kolloidchemische Beihefte*, **31**, 434 (1930); "The Surface Tension of Asphaltic Bitumens and Allied Substances at Various Temperatures" by F. J. Nellensteyn, Permanent Intern. Road Congr., Bull. No. 74, p. 58 (1931).

88 [p. 861] "Physical Properties of Coal Tars," by E. W. Volkmann and E. O. Rhodes, *J. Ind. Eng. Chem.*, **28**, 721 (1936).

89 [p. 862] "An Interfacial Tensionmeter for Universal Use," by P. L. du Noüy, *Biochem. Z.*, **155**, 113 (1925); *J. Gen. Physiol.*, 7, 625 (1925); "Determination of Surface-tension by the Ring Method (Torsion Balance)," by P. L. du Noüy, *Biochem. Z.*, **155**, 113 (1925); "Interfacial Tension Between Asphaltic Materials and Various Aqueous Solutions," by R. N. Traxler and C. U. Pittman, *J. Ind. Eng. Chem.*, **24**, 1003 (1932); "Theoretical Aspect of the Relation of Bitumen to Solid Matter," by F. J. Nellensteyn, World Petroleum Congr., London (Jul. 19–25, 1933); "Die Ausbreitung von Wasser auf Asphaltbitumen und Teer," by F. J. Nellensteyn and N. M. Roodenburg, *Kolloid-Zeitsch.*, **63**, 339 (1933); "The Surface Tension of Asphalt Bitumen," by R. N. J. Saal, *Chem.-Weekblad*, **32**, 486 (1935).

90 [p. 863] "Tentative Symbols for Heat Transmission" (C 108–34T), A.S.T.M. Tentative Standards 1936, 460; "Tentative Method of Test for Comparing the Thermal Conductivities of Solid Electrical Insulating Materials" (D 325–31T), A.S.T.M. Tentative Standards 1936, 1010.

91 [pp. 866, 870, 871] "Thermal Properties of Petroleum Products," Miscellaneous Publication No. 97, Bureau of Standards, Wash., D. C. (1929); "Volume Correction Table for Petroleum Oils," American Standard A.S.A. No: Z 11a-1928, American Standards Association.

92 [p. 866] "Tentative Method of Test for Comparing the Thermal Conductivities of Solid Electrical Insulating Materials" (D 325–31T), A.S.T.M. Tentative Standards 1936, 1004.

93 [p. 870] "Specific Heat of Some Crude Oils and their Residues when Topped to 100° C.," by H. R. Lang and J. Just, *Petroleum Tech.*, **17**, 585 (1931).

94 [p. 875] "Bestimmung des kubischen Ausdehnungskoeffizienten von Trinidad-Asphalt," by Heinrich Mallison, *Teer u. Bitumen*, **30**, 161 (1932).

95 [p. 875] "The Coefficient of Expansion of Tar," by J. M. Weiss, *J. Franklin Inst.*, **172**, 277 (1911); *A.G.A. Monthly*, **3**, 497 (1911); "Temperature Coefficient of Expansion of Petroleum Residuums," by H. Rossbacher, *J. Ind. Eng. Chem.*, **7**, 577 (1915); "Some Applications of Physical Chemistry in the Coal-tar Industry," by W. J. Huff, *J. Ind. Eng. Chem.*, **10**, 1016 (1918); "Volume-temperature Correction for Creosote Oil Measurements," by S. R. Church and J. M. Weiss, Proc. Am. Wood Pres. Assoc., p. 160 (1918).

96 [pp. 875 and 886] "Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation" (D 176–36T), A.S.T.M. Tentative Standards 1936, 871.

97 [p. 885] "Methods for Testing Coal Tar and Refined Tars, Oils and Pitches Derived Therefrom," by S. R. Church, *J. Ind. Eng. Chem.*, **3**, 227 (1911); "Chem. tech. Untersuchungsmethoden," by Heinrich Mallison, Vol. III, 295, Lunge-Berl, Germany (1912); "Apparatus for Determination of Breaking Point of Pitches," by H. E. Lloyd and P. P. Sharples, Proc. A.S.T.M., **19**, II, 782 (1919).

98 [p. 886] "Machine for the Determination of the Pliability of Prepared Roofing and the Breaking Point of Bitumens," by C. S. Reeve and F. W. Yeager, *Proc. A.S.T.M.*, **25**, II, 385 (1925).

99 [p. 886] "Eine neue Methode zur Feststellung des Brechpunktes bituminöser Bindemittel und bituminöser Massen," by Ing. Fraass, *Asphalt u. Teer Strassenbautech.*, **13**, 367 (1930); *Asphalt u. Teer*, **30**, 367 (1930); "Ueber die Bestimmung des Brechpunktes," by J. Miegl, *Strassenwesen*, **7**, Jul. 1931; "New Methods of Investigation for Characterizing the Solidification Point," by P. Herrmann, *World Petroleum Congr.* (London), **2**, 590 (1933); *Asphalt u. Teer Strassenbautechnik*, **33**, 391 (1933); "The Brittle Point of Bituminous Road Materials," by W. E. Golding and F. M. Potter, *Chem. & Ind.*, **53**, 628 (1934); "Bestimmung der Sprödigkeits-Temperature," by F. C. Van Heurn, *Archiv fur Technisches Messen*, May 1935; **Ger. Pat.** Design 1112027 of Feb. 4, 1930 to Paul Gebhardt Söhne.

100 [p. 889] "Untersuchungen über die Viskosität bituminöser Stoffe und deren gesetzmäszige Zusammenhänge," by K. A. Hoepfner, *Techn. Gemeindeblatt*, **6**, 75 (1929); "Die Viscositätszahlen bitumimöser Stoffe und deren gesetzmaszige Beziehungen untereinander," by Hans Metzger, *Z. angew. Chem.*, **43**, 289 (1930); "Starrpunkt und Viskosität bituminöser Stoffe," by Hans Metzger, Verlag von Wilhelm Knapp, Halle (Saale), (1930); **Ger. Pat.** Design 1099583 of Oct. 8, 1929 to Hans Metzger.

101 [p. 898] "Zur Untersuchung von Briquettepech," by J. Klimont, *Z. angew. Chem.*, **13**, 761 (1900); "A Melting-point Bridge for Petroleum Shale Oils and Shale Oil Distillates," by L. C. Karrick and V. F. Parry, *J. Ind. Eng. Chem.*, **15**, 600 (1923).

102 [p. 898] "New Method for Determining the Melting Point of Pitch," by H. F. French, *Analyst*, **37**, 62 (1912).

103 [p. 898] "Methods for Testing Coal Tar and Refined Tars, Oils and Pitches Derived Therefrom," by S. R. Church, *J. Ind. Eng. Chem.*, **3**, 230 (1911); "An Apparatus for Determining the Melting-Point of Substances of Indefinite Melting-Point," by A. P. Bjerregaard, *J. Ind. Eng. Chem.*, **5**, 938 (1913).

104 [p. 898] "Valuation of Coal-tar Pitch," by F. Muck, *Z. anal. Chem.*, **29**, 103 (1890); "A Comparison Method for Determining the Melting Point of Asphalt," by C. F. Mabery and O. J. Sieplein, *J. Am. Chem. Soc.*, **20**, 16 (1901); "The Softening-Point of Pitch," by P. E. Spielmann and G. C. Petrie, *J. Soc. Chem. Ind.*, **38**, 68T (1919).

105 [p. 898] "The Distinction between Coal-tar and Blast-furnace Pitches," by D. G. Buchanan, *J. Soc. Chem. Ind.*, **13**, 1098 (1894); "Investigations on Asphalt," by Jenö Kovacs, *Chem. Rev. Fett- u. Harz-Ind.*, **9**, 156 (1902); *J. Soc. Chem. Ind.*, **21**, 1077 (1902); "Apparatus for Determining Melting-points of Fats," by Leo Ubbelohde, *Z. angew. Chem.*, **15**, 1220 (1902); **18**, 1220 (1905); *Mitteilungen*, **22**, 203 (1904); "Untersuchung der Kohlenwasserstofföle und Fette," by David Holde, pp. 55–6 (1913); "Determination of the Melting-Point of Asphalt,"

by L. Schanin, *Z. angew. Chem.*, **27**, 215 (1914); "Notes on Oil Pitch," by W. Mansbridge, *J. Soc. Chem. Ind.*, **37**, 183T (1918); "Device for Determining the Dropping-Point (Melting-Point) of Fats, Waxes, Paraffin Wax, etc., and Especially Bitumens," by F. Dupré, *Chem.-Ztg.*, **42**, 398 and 445 (1918); "Determination of the Softening-Point of a Pitch or Bitumen," by G. M. Green, *J. Soc. Chem. Ind.*, **48**, 114T (1929); Apparatus manufactured by Bleckmann and Burger, Berlin, Auguststr. 3a.

106 [p. 898] "Determination of the Softening Point of Asphaltum and Other Plastic Substances," by D. F. Twiss and E. A. Murphy, *J. Soc. Chem. Ind.*, **38**, 405T (1919).

107 [p. 898] "Neuer Apparat zur Untersuchung von Paraffinen, Wachsen, Harzen, Pechen und Asphalten," by Anon., *Chem.-Ztg.*, **46**, 386 (1922); *J. Soc. Chem. Ind.*, **41**, 443A (1922); Apparatus marketed by C. Gerhardt, Bonn a. Rhein (Germany) under the name Nashan's "Malakograph."

108 [p. 898] **U. S. Pat.** 1291409 of Jan. 14, 1919 to L. W. Chubb and R. de S. Brown.

109 [p. 898] "Studien über die Steinkohlenbriquettierung, bei welcher Pech als Bindemittel angewendet wird," by E. Schenk, *Z. angew. Chem.*, **3**, 704 (1890); *Oesterr. Zeit. für Berg- u. Hüttenw.*, **38**, 463 (1890); "Determination of the Melting-Point of Pitch, Asphalt and Similar Materials," by G. Krämer and C. Sarnow, *Chem. Ind.*, **26**, 55 (1903).

110 [p. 898] "Melting Point Determination of Asphalt," by B. M. Margosches, *Chem. Rev. Fett- u. Harz-Ind.*, **11**, 277 (1904); "Melting-Point Determination of Asphalt," by M. Wendriner, *Z. angew. Chem.*, **18**, 622 (1905); "Apparatus for Determining the Melting Point of Asphalt," by Hugo Bauer, *Chem. Zeit.*, **29**, 382 (1905); *Chem. Rev. Fett- u. Harz-Ind.*, **12**, 258 (1905); "Determination of the Melting-Point of Asphalt," by Edmund Graefe, *Chem. Zeit.*, **30**, 298 (1906); "Melting Point of Asphalt," by E. Kolbe, Inaug. Dissert., Zurich (1908); "A Modification of the Krämer-Sarnow Apparatus for Determination of the Melting Point of Asphalt," by S. Thieberger-Campina, *Petroleum*, **3**, 896 (1907–8); "Melting Point Determination of Artificial Asphalt," by H. Offermann, *Petroleum*, **6**, 2117 (1910); *Z. angew. Chem.*, **24**, 2031 (1911); "Determination of the Softening Point of Asphalt," by L. Barta, *Petroleum*, **7**, 158 (1911); "Determination of the Softening Point of Pitch," by V. Abeles, *Chem. Zeit.*, **38**, 249 (1914); *Petroleum*, **11**, 160 (1915); "Determination of the Softening Point of Pitch," by M. Klinger, *Chem.-Ind.*, **37**, 220 (1914); *Analyst*, **39**, 136 and 321 (1914). *Chem. Zeit.*, **38**, 63 (1914); "Determination of the Softening Point of Pitches," by A. W. Jones, Jr., *Chem. Analyst*, **20**, 23 (1917); "Zur Bestimmung des Tropfpunktes von Fetten, Wachsen, Paraffinen, usw., inbesondere von Bitumen," by F. Simon, *Chem.-Ztg.*, **45** 1005 (1921); "Determination of the Melting-Point of Asphalts by the Krämer-Sarnow Method," by Anon., *Mat. grasses*, **13**, 5934 (1921); "A New Method for Determining the Melting Point of Pitches, Asphalts, etc.," by F. G. Hoffmann, *Brennstoff-Chem.*, **7**, 218 (1926); *Teer u. Bitumen*, **24**, 572 (1926); "The Softening Point of Pitch and Asphalt, According to G. Krämer and C. Sarnow," by Heinrich Mallison, *Z. angew. Chem.*, **40**, 927 (1927); "Apparatus for Determining the Softening Point of Paraffine Wax, Pitch, etc.," by H. Herbst, *Petróleum*, **25**, 1079 (1927); *Chem.-Ztg.*, **51**, 141 (1927); "Water Instead of Mercury in the Krämer-Sarnow Method," by Theodor Heydecke, *Teer u. Bitumen*, **26**, 567 (1928); "The Determination of the Softening Point of Pitch and Asphalts," by A. Spilker, *Z. angew. Chem.*, **42**, 263 (1929); "Determination of Melting Points of Pitches and Asphalts by the Krämer-Sarnow Method," by David Holde, *Petroleum Z.*, **25**, 411 (1929); "Determination of the Softening Points of Asphalts and Pitches by the Krämer-Sarnow Method," by Hugo Burstin, *Petroleum Z.*, **26**, 789 (1930); "Zur Bestimmung des Erweichungspunktes von Pechen," by H. Trutnovsky, *Teer u. Bitumen*, **28**, 362 (1930); "Accuracy of the Determination of the Softening Point of Bitumen by the Krämer-Sarnow-Barta Method," by Kornel Krell, *Asphalt u. Teer Strassenbautech.*, **32**, 89 (1932).

111 [p. 898] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, *Proc. A.S.T.M.*, **9**, 575 (1909); **11**, 679 (1911).

112 [p. 901] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, *Proc. A.S.T.M.*, **9**, 577 (1909).

113 [p. 901] Private communication from Mr. Dozier Finley, Emeryville, Cal.

114 [p. 903] "Improved Instruments for the Physical Testing of Bituminous Materials," by Herbert Abraham, *Proc. A.S.T.M.*, 11, 674 (1911).

115 [p. 904] "The Ring-and-Ball Method of Test for Softening-Point of Bituminous Materials, Resins and Similar Substances," by P. H. Walker, Bureau of Standards Research Paper No. 142, Wash., D. C. (Feb., 1930); *Bureau of Standards J. of Research*, 4, (Feb. 1930); "Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method)" (D 36–26), A.S.T.M. Standards 1936, II, 1098; "Ueber die Prüfung von Fugenvergussmassen auf Standfestigkeit in der Wärme durch Bestimmung des Erweichungspunktes nach Ring und Kugel," by R. Wilhelmi, *Bitumen*, 6, 135 (1936).

116 [p. 904] "Notes on the Ring-and-Ball Method," by F. C. Eaton, *J. Rheology*, 2, 392 (1931).

117 [p. 906] "The Ring and Ball Method of Test for Softening Point of Bituminous Materials, Resins and Similar Substances," by P. H. Walker, *Bureau of Standards J. of Research*, 4, 195 (1930); "Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus" (E 28–36T), A.S.T.M. Tentative Standards 1936, 1300.

118 [p. 908] "The Difference Between Krämer-Sarnow and Ring-and-Ball Values," by Heinrich Mallison, *Teer u. Bitumen*, 26, 602 (1928).

119 [p. 908] "Melting Point of Asphalt," by L. M. Proctor, *Chem. Met. Eng.*, 21, 81 (1910).

120 [p. 908] "Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method)" (D 61–24), A.S.T.M. Standards 1936, II, 1103.

121 [p. 911] "Methods for Determining the Melting-point of Asphalts," by J. G. Miller and P. P. Sharples, *Proc. A.T.S.M.*, 14, II, 503 (1914); *Chem. Eng.*, 20, 217 (1914); "Report of Sub-committee on Softening Point," *Proc. A.S.T.M.*, 15, Part I, 341 (1915).

122 [p. 911] "Relation Between the Melting-point and the Viscosity of Refined Tars," by P. P. Sharples, *J. Ind. Eng. Chem.*, 6, 285 (1914).

123 [p. 911] "Some Relations Between the Characteristics of Straight Distilled Tar Residues," by J. W. Kennedy, *Proc. A.S.T.M.*, 23, II, 341 (1923).

124 [p. 912] "The Softening-point of Plastic Materials Determined by the Widney Resiliometer and Expressed Graphically," by Alan Speedy, *J. Soc. Chem. Ind.*, 39, 18T (1920).

125 [p. 913] "Indentation of Asphalt Tile," by J. W. McBurney, *Proc. A.S.T.M.*, 34, Part II, 591 (1934).

126 [p. 913] "Standard Method of Test for Melting Point of Petrolatum" (D 127–30), A.S.T.M. Standards 1936, II, 942; American Standard A.S.A. No: Z 11.22–1932 American Standards Association; American Petroleum Institute Standard A.P.I. No: 523–30.

127 [p. 914] "Paraffin Wax and Its Properties, Methods of Testing, etc.," Bureau of Mines Technical Paper No. 368, Wash., D. C. (1925); "Standard Method of Test for Melting Point of Paraffin Wax" (D 87–22), A.S.T.M. Standards 1936, II, 938; American Standard A.S.A. No: Z 11.4–1928 American Standards Association; American Petroleum Institute Standard A.P.I. No: 513–29.

128 [p. 917] "Investigation of Paraffin, Stearin and Similar Substances," by Franz Dangl, *Petroleum Z.*, 27, 142 (1931).

129 [p. 917] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 567, John Wiley & Sons, New York (1908); "Fugenausgussmassen für Strassendecken aus Zementbeton," by F. Seufert, *Bitumen*, 6, 31 (1936).

130 [p. 917] Deutscher Normenausschuss DIN-1995 and DIN-1996, Heft, 1, page 13, Beuth-Verlag, Berlin (March 1934).

131 [p. 918] "The Evaluation of Pitch," by H. F. Taylor, *J. Soc. Chem. Ind.*, 45, 417T (1926); "Standard Methods for Testing Tar and Its Products," p. 192, Standardization of Tar Products Tests Committee, London (1929).

132 [p. 921] "Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds" (D 6–33), A.S.T.M. Standards 1936, II, 1071.

133 [p. 924] D. I. N. 1995 and 1996; Heft 1, p. 14 (Jan., 1931).

134 [p. 924] "Standard Method of Test for Residue of Specified Penetration" (D 243–36),

A.S.T.M. Standards 1936, II, 1083; American Assoc. State Highway Officials Standard A.A.S.H.O. No: T-56.

135 [p. 928] "Distillation of Tar," by P. P. Sharples, *J. Ind. Eng. Chem.*, **5**, 466 (1913).

136 [p. 928] "Analytical Distillation of Coal-tar," by E. B. Kester, W. D. Pohle and L. P. Rockenbach, Dept. of Investigations No. 3171, U. S. Bureau of Mines, Wash., D. C. (Apr., 1932); "Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment" (D 20–30), A.S.T.M. Standards 1936, II, 1053.

137 [p. 930] "Standard Method of Test for Distillation of Creosote" (D 246–33), A.S.T.M. Standards 1936, II, 542; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-62.

138 [p. 931] "Specifications for Liquid Asphaltic Road Materials," The Asphalt Institute, New York (Jan. 1933); "Standard Method of Test for Separation of Liquid Asphaltic Products" (D 402–36), A.S.T.M. Standards 1936, II, 1065; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-78.

139 [p. 935] "Flash-Point of Oil," Bureau of Mines Tech. Paper No. 49, Wash., D. C. (1913).

140 [p. 935] "Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester" (D 93–36), A.S.T.M. Standards 1936, II, 897; American Standard A.S.A. No: Z 11.7–1936 American Standards Association; American Petroleum Institute Standard A.P.I. No: 510–36; American Association State Highway Officials' Standard A.A.S.H.O. No: T-73.

141 [p. 941] "Flash-point of Oils," by I. C. Allen and A. S. Crossfield, Tech. Paper No. 49, Petroleum Technology 10, Dept. of Interior, Bureau of Mines, Wash., D. C. (1913).

142 [p. 941] "Standard Method of Test for Flash and Fire Points by Means of Open Cup" (D 92–33), A.S.T.M. Standards 1936, II, 892; American Standard A.S.A. No: Z 11.6–1933 American Standards Association; American Petroleum Institute Standard A.P.I. No: 511–33.

143 [p. 945] "Standard Method of Test for Flash Point by Means of the Tag Closed Tester" (D 56–36), A.S.T.M. Standards 1936, II, 907; American Standard A.S.A. No: Z 11.24–1936 American Standards Association; American Petroleum Institute Standard A.P.I. No: 509–36.

144 [p. 949] "Standard Methods of Laboratory Sampling and Analysis of Coal and Coke" (D 271–33), A.S.T.M. Standards 1936, II, 387; American Standard A.S.A. No: K 18–1933 American Standards Association.

145 [p. 949] "The Test for Volatile Combustible Matter as Applied to Coal-tar Pitch," by S. R. Church and W. H. Fulweiler, *Proc. A.S.T.M.*, **28**, II, 848 (1928).

146 [p. 949] "The Volatile Combustible Matter of Coal-tar Pitch," by J. M. Weiss, *Proc. A.S.T.M.*, **32**, Part II, 676 (1932).

147 [p. 949] "Standard Method of Test for Coke Residue of Creosote" (D 168–30), A.S.T.M. Standards 1936, II, 553; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-61.

148 [p. 951] "A Study of Certain Methods for Determining Total Soluble Bitumen in Paving Materials," by S. Avery and R. Corr, *J. Am. Chem. Soc.*, **28**, 648 (1906); "The Proximate Composition of Physical Structure of Trinidad Asphalt, with Special Reference to the Behavior of Mixtures of Bitumen and Fine Mineral Matter," by Clifford Richardson, *Proc. A.S.T.M.*, **6**, 509 (1906); "The Determination of Soluble Bitumen," by Prévost Hubbard and C. S. Reeve, *Proc. A.S.T.M.*, **10**, 420 (1910); "The Bitumen Content of Coarse Bituminous Aggregates," by Prévost Hubbard, *Proc. Intern. Assoc. Testing Materials*, XXV-2 (1912).

149 [p. 952] "Standard Method of Test for the Determination of Bitumen" (D 4–27), A.S.T.M. Standards 1936, II, 1041; American Standard A.S.A. No: A 37.3–1930 American Standards Association.

150 [p. 955] "Estimation of Moisture in Cold Mixes," by A. R. Ebberts, 10th Annual Paving Conference, pp. 343–5 (1932).

151 [p. 956] "Analysis of Calcareous Asphaltum and Paving Mixtures," by A. Prettner, *Chem. Zeit.*, 33, 917 and 926 (1909).

152 [p. 957] "Synthetic Asphaltic Limestone, with Special Reference to the Process of Zimmer," by H. Kröhnke, *Chem. Zeit.*, **44**, 253 (1920).

153 [p. 958] "Influence of Heat and of Very Fine Mineral Admixtures on the Characteristics of Bituminous Road Binders," by Knipping and Bewersdorf, *Asphalt u. Teer Strassenbautech.*, **31**, 699 (1931).

154 [p. 958] "Beitrag zur Analyse von Bitumen- und Teer-Mineralgemischen," by H. Nüssel and H. Henneke, *Bitumen*, **5**, 143 (1935).

155 [p. 959] "The Manufacture and Testing of Asphalt Paving Materials," by D. M. Wilson, *J. Soc. Chem. Ind.*, **51**, 61, (1932).

156 [p. 959] "The Recovery and Examination of the Asphalt in Asphaltic Paving Mixtures," by J. H. Bateman and Charles Delp, *Proc. A.S.T.M.*, **27**, II, 465 (1927); "The Effect of the Mixing Temperature on the Physical Properties of an Oil Asphalt in a Sheet Asphalt Mixture," by J. H. Bateman and H. L. Lehmann, *Proc. A.S.T.M.*, **29**, II, 943 (1929).

157 [p. 959] "Neue Methode zur Extraktion von Asphalt aus Gemischen mit Gestein oder aus Emulsionen, bei der die Aenderung der Viskositätskennzahlen vermieden Wird," by K. A. Hoepfner and Hans Metzger, *Strassenbau*, **15** (May 20, 1930).

158 [p. 962] "Recovery of Asphaltic Bitumens from Solutions, as far as Possible without Altering their Properties," by Jacques Greutert, *J. Inst. Petroleum Tech.*, **18**, 846 (1932); "Method and Apparatus for the Recovery of Asphalt," by Gene Abson, *Proc. A.S.T.M.*, **33**, II, 704 (1933).

159 [p. 962] "New Process for the Investigation of Bitumen and Bitumen-Containing Mixtures," by Jacques Greutert, *Bitumen*, **3**, 49 (1933); "The Recovery of Bitumen," by L. J. Chalk, *J. Soc. Chem. Ind.*, **56**, 156T (1937).

160 [p. 962] "Analysis of Asphalt Limestone and Asphalt Powder," by Meyer Prettner, *Chem.-Ztg.*, **33**, 917 and 926 (1909).

161 [p. 962] "Carbon Tetrachloride and Its Use as a Solution for Differentiating Bitumens," by Clifford Richardson and C. N. Forrest, *J. Soc. Chem. Ind.*, **24**, 310 (1905); "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 546, John Wiley & Sons, New York (1908); "The Value of the Carbene Requirement in Asphalt Specifications," by Lester Kirschbraun, *Munic. Eng.*, **35**, 349 (1909); "On the Formation of Carbenes," by D. B. W. Alexander, *J. Ind. Eng. Chem.*, **2**, 242 (1910); "Studies on the Carbenes," by K. J. Mackenzie, *J. Ind. Eng. Chem.*, **2**, 124 (1910); "Some Experiments on Technical Bitumens," by S. R. Church and J. M. Weiss, *Proc. A.S.T.M.*, **15**, I, 274 (1915); "Some Relations of the Effect of Overheating to Certain Physical and Chemical Properties of Asphalts," by A. W. Hixson and H. E. Hands, *J. Ind. Eng. Chem.*, **9**, 651 (1917).

162 [p. 963] "Some Effects of Certain Solvents on Tars in 'Free Carbon' Determination," by G. S. Monroe and H. J. Broderson, *J. Ind. Eng. Chem.*, **9**, 1100 (1917); "Coke-Oven and By-Products Workers' Chemistry," by T. Biddulph-Smith, J. B. Lippincott Co., Phila., p. 47 (1921).

163 [p. 963] "Standard Method of Test for The Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride" (D 165–27), A.S.T.M. Standards 1936, II, 1044.

164 [p. 964] "Petroleum Oil and Coal," by Julius Marcusson, *Chem. Ztg.*, **42**, 437 (1918); *J. Soc. Chem. Ind.*, **39**, 98A (1919); "Paraffinic Petroleum Asphalt," by J. Müller and S. von Pilat, *Asphalt u. Teer Strassenbautech.*, **34**, 649 (1934).

165 [p. 964] "Plastic Properties of Asphaltic Bitumen," by R. N. J. Saal and G. Koens, *J. Inst. Petroleum Tech.*, **19**, 176 (1933).

166 [p. 965] "Determination of Asphalt in Lubricating Oils," by E. A. Evans, *J. Inst. Petroleum Tech.*, **9**, 384 (1923); "Analysis of Asphalt," by L. A. Linton, *J. Amer. Chem. Soc.* **16**, 808 (1894); "Determination of the Asphalt Content of Oils and Pitches," by F. Schwartz, *Chem. Ztg.*, **35**, 413 and 1417 (1911).

167 [p. 965] "Determination of Asphaltenes," by F. J. Nellensteyn and N. M. Roodenburg, *Chem.-Ztg.*, **54**, 819 (1930).

168 [p. 965] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 543, John Wiley & Sons, New York (1908); "Laboratory Manual of Bituminous Materials," by Prévost Hubbard, 1st Edition, p. 90, John Wiley & Sons, Inc., New York (1916).

169 [p. 966] "Socalled Free Carbon of Bituminous Coal-Tar," by Heinrich Mallison, *Asphalt u. Teer*, **35**, 1001 (1935).

170 [p. 966] "Free Carbon, Its Nature and Determination in Tar Products," by J. M. Weiss, *J. Ind. Eng. Chem.*, **6**, 279 (1914); "Some Effects of Certain Solvents on Tars in the 'Free Carbon' Determination," by G. S. Monroe and H. J. Broderson, *J. Ind. Eng. Chem.*, **9**, 1100 (1917); "Notes on Free Carbon in Tar," by J. M. Weiss and C. R. Downs, *J. Ind.Eng. Chem.*, **10**, 400 (1918); "The Influence of Carbonizing Conditions upon the Free Carbon Content of Tars," by H. Hollings, *J. Soc. Chem. Ind.*, **45**, 406T (1926).

171 [p. 966] "Free Carbon of Bituminous Coal Tar," by Julius Marcusson, *Chem.-Ztg.*, **54**, 795 (1930).

172 [p. 966] "Standard Method of Test for Insoluble Matter in Creosote" (D 367–33), A.S.T.M. Standards 1936, II, 535; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-81.

173 [p. 968] "Standard Method of Chemical Analysis of Rubber Products" (D 297-36), A.S.T.M. Standards 1936, II, 1249.

174 [p. 968] "Colloid Chemistry," by Jerome Alexander, Vol. III, Chapter on "The Free Carbon of Coal Tar," by J. M. Weiss and C. R. Downs, p. 527, The Chemical Catalog Co., Inc., New York (1931).

175 [p. 968] "Determination of Free Carbon in Tars, Pitches, and the Like," by Ernst Berl and H. Schildwächter, *Brennstoff-Chemie*, **9**, 137 (1928).

176 [p. 968] "The Determination of Benzol-Insoluble Free Carbon in Coal-tar Pitch," by Karl Frey, *Asphalt u. Teer Strassenbautech.*, **32**, 358 (1932).

177 [p. 969] "Physical Properties of Coal Tars," by E. W. Volkmann and E. O. Rhodes, *J. Ind. Eng. Chem.*, **28**, 721 (1936).

178 [p. 970] "Acetone as a Solvent for Bitumens," by Anon., *Oil Trade J.*, **13**, 123 (1922); *Chem. Met. Eng.*, **27**, 651 (1922); *Petroleum Age*, **10**, 92 (1922).

179 [p. 974] "Die Verfahren zur Ermittlung von Wasser und wasserdampfflüchtigen Lösemitteln in Teer und Oel," by Robert Kattwinkel, *Teer u. Bitumen*, **27**, 181, 201 and 217 (1929).

180 [p. 974] "Standard Method of Test for Water in Creosote" (D 370–33), A.S.T.M. Standards 1936, II, 538; American Assoc. State Highway Officials' Standard A.A.S.H.O. No: T-83.

181 [p. 975] "The Dehydration of Tar in the Laboratory," by W. J. Huff, *J. Ind. Eng. Chem.*, **13**, 1123 (1921).

182 [p. 975] "A Rapid Determination of Water in Tar," by P. Smit, *Chem. Weekblad*, **16**, 852 (1919).

183 [p. 975] "Methods for the Determination of Water in Petroleum and its Products," by I. C. Allen and W. A. Jacobs, Tech. Paper No. 25, Dept. of Interior, Bureau of Mines, Wash., D. C. (1912); "A Convenient Method for the Determination of Water in Petroleum and Other Organic Emulsions," by E. W. Dean and D. D. Stark, *J. Ind. Eng. Chem.*, **12**, 486 (1920).

184 [p. 975] "Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials" (D 95–30), A.S.T.M. Standards 1936, II, 997; American Standard A.S.A. No: Z 11.9–1930 American Standards Association; American Petroleum Institute Standard A.P.I. No: 519–30.

185 [p. 978] "Standard Method of Test for Water and Sediment in Petroleum Products, by Means of Centrifuge" (D 96–35), A.S.T.M. Standards 1936, II, 1001; American Standard A.S.A. No: Z 11.8–1935; American Petroleum Institute Standard A.P.I. No: 520–35.

186 [p. 984] "Methods of Analyzing Coal and Coke," by F. M. Stanton and A. C. Fieldner, Tech. Paper No. 8, p. 18, Dept. of Interior, Bureau of Mines, Wash., D. C. (1926); "Standard Methods of Laboratory Sampling and Analysis of Coal and Coke" (D 271–33), Section on "Methods of Analysis," A.S.T.M. Standards 1936, II, 396; American Standard A.S.A. No: K 18–1933 American Standards Association.

187 [p. 984] "Standard Method of Test for Sulphur in Petroleum Oils by Bomb Method" (D 129–34), A.S.T.M. Standards 1936, II, 974; American Standard A.S.A. No: Z 11.13–1934

American Standards Association; American Petroleum Institute Standard A.P.I. No: 516–34.

188 [p. 987] "Sulphur in Tar Residues," by Prévost Hubbard and C. S. Reeve, *Proc. A.S.T.M.*, **11**, 666 (1911); "The Sulphur Content of Fuels, and Especially Petroleum Products," by I. C. Allen and I. W. Robertson, Tech. Paper No. 26, Bureau of Mines, Dept. of Interior, Wash., D. C. (1912); "The Detection and Determination of Sulphur in Petroleum," by C. K. Francis and C. W. Crawford, *J. Ind. Eng. Chem.*, **9**, 479 (1917); "A New Method for the Determination of Sulphur in Oils," by C. E. Waters, *J. Ind. Eng. Chem.*, **12**, 482 (1920); "Sulphur in Petroleum Oils," by C. E. Waters, Tech. Paper No. 177, Bureau of Standards, Wash., D. C. (Oct. 20, 1920); "Standard Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline" (D 130–30), A.S.T.M. Standards 1936, II, 973; American Standard A.S.A. No: Z 11.21–1930 American Standards Association; American Petroleum Institute Standard A.P.I. No: Z 521–30; "Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method" (D 90–34T), A.S.T.M. Tentative Standards 1936, 643 American Tentative Standard A.S.A. No: Z 11.38-1935 American Standards Association.

189 [p. 987] "The Composition and Examination of Residues from Fat Distillation," by Julius Marcusson, *Z. angew. Chem.*, **24**, 1297 (1911).

190 [p. 988] "Methods of Analyzing Coal and Coke," by F. M. Stanton and A. C. Fieldner, Tech. Paper No. 8, Bureau of Mines, Dept. of Interior, Wash., D. C. (1926); "The Estimation of Nitrogen in Petroleum and Bitumens," by E. J. Poth, W. D. Armstrong, C. C. Cogburn and J. R. Bailey, *J. Ind. Eng. Chem.*, **20**, 831 (1928).

191 [p. 989] "Standard Methods of Laboratory Sampling and Analysis of Coal and Coke" (D 271–33), A.S.T.M. Standards 1936, II, Section on "Methods of Analysis," 396; American Standard A.S.A. No: K 18–1933 American Standards Association.

192 [p. 990] "Determination of Mean Molecular Weights of Lubricating Oils by Improved Cryoscopic Methods," by N. G. Gullick, *J. Inst. Petroleum Tech.*, **17**, 541 (1931).

193 [p. 990] "Colloid Chemistry of Asphalts," by C. Mack, *J. Physical Chem.*, **36**, 2901 (1932).

194 [p. 991] "Methods of Analysis Used in the Coal-tar Industry," by J. M. Weiss, *J. Ind. Eng. Chem.*, **10**, 1911 (1918).

195 [p. 993] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 558, John Wiley & Sons, New York (1908); "Untersuchung der Kohlenwasserstofföle und Fette," by David Holde, p. 45, Verlag von Julius Springer, Berlin (1913); "Laboratory Manual of Bituminous Materials," by Prévost Hubbard, p. 100, John Wiley & Sons, Inc., New York (1916).

196 [p. 993] "The Estimation of Wax in Pitch, by W. Littlejohn and W. H. Thomas, *J. Inst. Petroleum Tech.*, **16**, 814 (1930).

197 [p. 994] "Determination and Fractional Separation of Paraffin with Methyl Ethyl Ketone (Butanone)," by Fritz Schwarz, *Mitt. Materialprüfungsamt*, **36**, 241 (1918).

198 [p. 994] "Determination of Paraffin Wax in Crude Wax," by L. M. Henderson and S. W. Ferris, *J. Ind. Eng. Chem.*, **19**, 262 (1927).

199 [p. 994] "The Time Factor in the Dry Distillation of the Marcusson-Eickmann Paraffin Determination," by H. Suida and W. Janisch, *Asphalt u. Teer*, **31**, 503 (1931); "Water-White Hydrocarbons from Trinidad Asphalt," by H. T. Gaetz, p. 68, Grafton Publishing Corp., Los Angeles, Cal. (1935); "Water-White Hydrocarbons from Trinidad Asphalt," by H. T. Gaetz, *Refiner and Natural Gasoline Manufacturer* (Aug. 1936).

200 [p. 994] "A New Method for Determination of Paraffin in Asphalts," by Jakob Müller and Damian Wandycz, *Asphalt Teer Strassenbautech.*, **32**, 708 (1932); **33**, 421 (1933), **U. S. Pat.** 1278023 of Sep. 3, 1918 to R. R. Rosenbaum.

201 [p. 995] "Algebraic Method for the Detection of Adulteration of Ceresine with Paraffin," by H. Smelkus, *Chem. Zeit.*, **44**, 273 and 286 (1920); "Detection of Paraffin in Ceresine," by David Holde and K. H. Schünemann, *Z. angew. Chem.*, **41**, 368 (1928).

202 [p. 995] "Commercial Paraffin Waxes—Specific Refraction in Liquid and Solid States," by J. M. Page, Jr., *J. Ind. Eng. Chem.*, **28**, 856 (1936).

203 [p. 995] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 544, John Wiley & Sons, New York (1908).

204 [p. 996] "The Analysis and Grading of Creosotes," by A. L. Dean and Ernest Bateman, Forest Service Circular No. 112, Wash., D. C. (1908); "Modification of the Sulphonation Test for Creosote," by Ernest Bateman, Forest Service Circular No. 191, Wash., D. C. (1911); "Methods for Testing Coal Tar and Refined Tars, Oils and Pitches Derived Therefrom," by S. R. Church, *J. Ind. Eng. Chem.*, **3**, 233 (1911); **5**, 196 (1913); "Paraffin Bodies in Coal-Tar Creosote and Their Bearing on Specifications," by S. R. Church and J. M. Weiss, *J. Ind. Eng. Chem.*, **6**, 396 (1914).

205 [p. 996] "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, pp. 91 and 106, Verlag von Wilhelm Engelmann, Leipzig (1921); "Determination of Tar and Bitumen in Mixtures," by Julius Marcusson and Ph. Lederer, *Verkehrtechnik*, **8**, 121 (1928).

206 [p. 997] "Estimation of Tar and Bitumen in Mixtures," by Julius Marcusson and Ph. Lederer, *Petroleum Z.*, **26**, 866 (1930).

207 [p. 997] "Zur Bestimmung des Gehalts an Erdölasphalt in Gemischen mit Steinkohlenteer," by K. A. Hoepfner and Hans Metzger, *Tech. Gemeindeblatt*, **31**, 93 (1930).

208 [p. 999] "Analysis of Coal-tar-asphalt Mixtures," by Heinrich Mallison, *Asphalt u. Teer Strassenbautech.*, **30**, 183 (1930).

209 [p. 999] "Methods of Asphalt Examination," by Albert Sommer, *J. Ind. Eng. Chem.*, **2**, 181 (1910); "Application of the Dimethyl Sulphate Test for Determining Small Amounts of Petroleum or Asphalt Products in Tars," by C. S. Reeve and R. H. Lewis, 8th Intern. Congr. of Applied Chem., **25**, 727 (1912); *J. Ind. Eng. Chem.*, **5**, 293 (1913); "Distinction between Lignite- and Coal-tar Oil," by Anon., *Mitt. Materialprüfungsamt*, **42**, 67 (1925).

210 [p. 999] "Diethyl Sulfate in the Examination of Hydrocarbon Oils," by J. N. Taylor, *J. Ind. Eng. Chem.*, **19**, 76 (1927).

211 [p. 999] "Chemical Composition of Lubricating Oils," by A. M. Nastjukoff, *J. Russ. phys.-chem. Ges.*, 881 (1904); *Petroleum*, **4**, 1336 and 1397 (1909); "The Formolite Reaction of Nastukoff (A. M.) as Applied to Oil Residues and Natural Asphalts," by Clifford Richardson, *J. Ind. Eng. Chem.*, **8**, 319 (1916); *J. Soc. Chem. Ind.*, **23**, 1082 (1916); "Composition of Mineral Cylinder Oils," by Julius Marcusson, *Mitt. Materialprüfungsamt*, **40**, 308 (1922).

212 [p. 1000] "Low-temperature Ductility of Filler-grade Asphalts," by W. F. Smith, *Proc. A.S.T.M.*, **27**, II, 476 (1927).

213 [p. 1000] "Degree of Mercuration of Crude Petroleum Oils and their Products for the Control of the Refinery Processes," by Jenö Tausz, *Petroleum Z.*, **13**, 649 (1918); *Z. angew. Chem.*, **31**, 336 (1918); *J. Soc. Chem. Ind.*, **37**, 554A (1918) and **38**, 4A (1919).

214 [p. 1000] "Test for Coal-tar Pitch in Natural Asphaltum, Petroleum Asphalt, and in the Presence of Colophonium," by F. J. Nellensteyn and J. C. M. Sauerbier, *Z. angew. Chem.*, **42**, 722 (1929).

215 [p. 1002] "Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants" (D 188–27T), A.S.T.M. Tentative Standards 1936, 629; American Tentative Standard A.S.A. No: Z 11.12–1928, American Standards Association; "Proposed Method of Test for Neutralization Number of Petroleum Products and Lubricants (Potentiometric Method)" (Appendix V), *Proc. A.S.T.M.*, **36**, Part I, 409 (1936).

216 [p. 1003] "Ueber Montanwachs und eine neue Methode zur Bestimmung der Säuerzahl und Verseifungszahl dunkel gefärbter Oele, Fette und Wachse," by R.. Pschorr, J. K. Pfaff and W. Berndt, *Z. angew. Chem.*, **34**, 334 (1921); *J. Soc. Chem. Ind.*, **40**, 593A (1921); "Standard Method of Test for Saponification Number" (D 94–36), A.S.T.M. Standards 1936, II, 970; American Standard A.S.A. No: Z 11.20–1936 American Standards Association; American Petroleum Institute Standard A.P.I. No: 514–36; "Tentative Method of Test for Saponification Number of Electrical Insulating Oils (Modified Baader Method)" (D 438–36T) A.S.T.M. Tentative Standards 1936, 984.

217 [p. 1003] "Butyl Alcohol as a Medium for the Determination of Saponification Numbers," by A. M. Pardee, R. L. Hasche and E. E. Reid, *J. Ind. Eng. Chem.*, **12**, 481 (1920).

218 [p. 1003] "Potentiometric Determination of Saponification Number of Mixtures of

Asphalt and Drying Oils," by J. V. Demarest and William Rieman, 3rd, *J. Ind. Eng. Chem.*, Analytical Edit., **3**, 17 (1931).

219 [p. 1004] "Saponification Numbers of Asphaltic Petroleum Residues," by J. H. Bruun and L. W. Claffey, *J. Ind. Eng. Chem.*, Analytical Edition, **8**, 255 (1936).

220 [p. 1005] "Standard Method of Analysis of Grease" (D 128–27), A.S.T.M. Standards 1936, II, 926; American Standard A.S.A. No: Z 11.16–1928, American Standards Association; American Petroleum Institute Standard A.P.I. No: 501–29.

221 [p. 1007] "Analysis of Crude Glycerin," by International Standard Methods, *J. Soc. Chem. Ind.*, **30**, 556 (1911); "Aids in the Commercial Analysis of Oils, Fats and Their Commercial Products," by G. F. Pickering, Chas. Griffin & Co., Ltd., London (1917).

222 [p. 1007] "Zur Unterscheidung von Natur- und Kunst-Asphalt," by Julius Marcusson, *Z. angew. Chem.*, **29**, I, 21 (1916); Les Matières Grasses, **9**, 4490 (1916); "Standards for Products of the Ashpalt Industry," by Julius Marcusson, *Mitt. königl. Materialprüfungsamt*, **34**, 40 (1916); "Die chemische Aufbau der Naturasphalte," by Julius Marcusson, *Z. angew. Chem.*, **29**, 346 (1916); *Mitt. königl. Materialprüfungsamt*, **36**, 209 (1918); "Chemical Composition of Artificial Asphalts," by Julius Marcusson, *Z. angew. Chem.*, **31**, 113 and 119 (1918); "Relation between Petroleum and Bituminous Coal," by Julius Marcusson, *Mitt. Materialprüfungsamt*, **37**, 27 (1919); *Z. angew. Chem.*, **32**, 113 (1919); "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, pp. 21–29, Verlag von Wilhelm Engelmann, Leipzig (1921); "Asphaltenes and Petroleum Resins in Petroleum and Petroleum Products," by A. Sachanen and N. Wassiliew, *Petroleum*, **36**, 1618 (1927); "Asphaltenes and Asphaltic Resins," by M. Naphtali, *Asphalt u. Teer Ind. Zeit.*, **9**, 225 (1928); "Chemical Composition of Asphalts and Asphaltic Materials," by V. Kalichevsky and S. C. Fulton, *Nat. Petroleum News*, **23**, 33 (Dec. 23, 1931); "Determination of Hard and Soft Asphalts, Resins, Paraffin-free Oil, and Paraffin in Bitumens," by W. Maass, *Petroleum Z.*, **28**, No. 21, 1 (1932).

223 [p. 1007] "Mineral Oil Asphalts," by Hans Pöll, *Petroleum*, **28**, No. 36 (1932); *Asphalt u. Strassenbau*, p. 2 (1932).

224 [p. 1007] "The Colloidal Structure of Asphalt, by R. Wilhelmi, *Erdöl u. Teer*, **8** 416 (1932).

225 [p. 1007] "Determination of Hard Asphalt, Soft Asphalt, Asphaltic Resin and Refined Oily Constituents, Including Paraffin," by W. Maass, World Petroleum Congress (London), **2**, 557 (1933).

226 [p. 1010] "The Vanillin-Hydrochloric Acid Reaction," by Leopold Rosenthaler, *Z. analyt. Chem.*, **44**, 292 (1905).

227 [p. 1013] "Distinction between Lignite Pitch and Other Pitches," by Edmund Graefe, *Chem.-Zeit.*, **30**, 298 (1906); "Natural and Artificial Asphalt," by Julius Marcusson and R. Eichmann, *Chem.-Zeit.*, **32**, 965 (1908); "A New Determination of Tar in Bitumen-Tar Mixtures," by P. Kaunert and E. Florian, *Oel, Kohle, Erdöl u. Teer*, **12**, 230 (1936).

228 [p. 1013] "Examination of Asphalt," by Julius Marcusson, *Chem. Rev. Fett- u. Harz-Ind.*, **18**, 47 (1911); *Mitt. königl. Materialprüfungsamt*, **38**, 79 (1920).

229 [p. 1014] "Analyses of Asphaltic Bitumen-Tar Mixtures," by F. J. Nellensteyn, World Petroleum Congr., London (Jul. 19–25, 1933).

230 [p. 1016] "Quantitative Estimation of Colophony in Residues of Coal-Tar Distillates," by David Holde and F. Meister, *Chem. Ztg.*, **35**, 793 (1911).

CHAPTER XXXIII

1 [p. 1017] "Federal Spec. for Road and Paving Materials; General Specifications (Methods for Sampling and Testing)" SS-R-406, Federal Standard Stock Catalogue, Section IV, (Part 5), Nov. 8, 1935.

2 [p. 1017] "Ein schnelles Verfahren zur Untersuchung von Proben bituminöser Beläge," by Ib Hvidberg, *Bitumen*, **6**, 169 (1936).

3 [p. 1018] "The Action of Water on Asphalts," by G. C. Whipple and D. D. Jackson, *Eng. News*, **43**, 187 (1900); "Testing of Bitumens for Paving Purposes," by A. W. Dow,

Proc. A.S.T.M., 3, 368 (1903); *Eng. News*, 51, 520 (1904); "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, p. 461, John Wiley & Sons, New York (1908).

4 [p. 1018] "Adhesion Tension in Asphalt Pavements; its Significance and Methods Applicable in its Determination," by Victor Nicholson, *Proc. Assoc. Asphalt Paving Technologists*, p. 28, Detroit, Mich., (Jan. 14, 1932); "Adhesion in Relation to Bituminous Road Materials," by A. R. Lee, *J. Soc. Chem. Ind.*, 55, 23T (1936); "Zur Frage der Haftfestigkeit," by Wilhelm Geissler, *Teer u. Bitumen*, 34, 415 (1936).

5 [p. 1019] "The Manufacture and Testing of Asphalt Paving Materials," by D. M. Wilson, *J. Soc. Chem. Ind.*, 51, 61 (1932); "Compression Testing of Asphalt Paving Mixtures," by Roland Vokac, *Proc. A.S.T.M.*, 36, II, 552 (1936).

6 [p. 1020] "Suggested Procedure in Design of Sheet Asphalt," by I. Bencowitz, *J. Ind. Eng. Chem.*, 29, 98 (1937).

7 [p. 1020] "A Practical Method for Determining the Relative Stability of Fine-Aggregate Asphalt Paving Mixtures," by Prévost Hubbard and F. C. Field, *Proc. A.S.T.M.*, 25, II, 335 (1925); "A Stability Test for Bituminous Paving Mixtures," by W. J. Emmons and B. A. Anderton, *Proc. A.S.T.M.*, 25, 346 (1925); "A Study of Certain Factors Affecting the Stability of Asphalt Paving Mixtures," by Prévost Hubbard and F. C. Field, *Proc. A.S.T.M.*, 26, II, 577 (1926); "The Manufacture and Testing of Asphalt Paving Mixtures," by D. M. Wilson, *J. Soc. Chem. Ind.*, 51, 61 (1932); "Asphalt Paving Mixtures," by The Aphalt Institute, New York (1930); "Bituminous Plastics—Determination of Flow Properties," by R. N. Traxler, *J. Ind. Eng. Chem.* (Analyt. Ed.), 8, 187 (1936); **U. S. Pats.** 1826732 of Oct. 13, 1931 to G. E. Chatillon; 2076592 of Apr. 13, 1937 to E. O. Rhodes.

8 [p. 1020] "Adaptation of the Stability Test to Include Coarse Aggregate Asphalt Mixtures," by Prévost Hubbard and F. C. Field, *Proc. Technical Session Assoc. Asphalt Paving Technologists*, p. 109, Detroit, Mich. (Jan. 14, 1932); "The Design and Testing of Asphalt Paving Mixtures," by N. H. Taylor, *J. Soc. Chem., Ind.*, 51, 415T (1932); "The Strength of Asphalt Mixtures," by H. T. Horsefield, *J. Soc. Chem. Ind.*, 53, 107T (1934); "The Mechanical Testing of Plastic Road Materials," by B. G. Manton and W. G. Wren, *J. Soc. Chem. Ind.*, 53, 141T (1904).

9 [p. 1022] "A Stability Test for Bituminous Paving Mixtures," by W. J. Emmons and B. A. Anderton, *Proc. A.S.T.M.*, 25, 346 (1925).

10 [p. 1022] **U. S. Pat.** 1979267 of Nov. 6, 1934 to H. L. Howe.

11 [p. 1022] L. W. Page, Bull. No. 79, Bureau of Chem., U. S. Dept. of Agri., Wash., D. C. (1908); "The Physical Testing of Rock for Road Building," by A. T. Goldbeck and F. H. Jackson, Bull. No. 44, Office of Public Roads, U. S. Dept. of Agri., Wash., D. C. (Jun. 10, 1912).

12 [p. 1022] "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, pp. 428 and 585, John Wiley & Son, New York (1908).

13 [p. 1023] "The Asphalt Association," Circular No. 10, New York (1921).

14 [p. 1024] "Ueber das Verhalten von Bitumen und Asphalt-mischungen bei Stossbeanspruchungen," by J. P. Pfeiffer, D. Oosterhof and J. van den Berge, *Bitumen*, 6, 25 (1936).

15 [p. 1025] "Die Natürlichen und Künstlichen Asphalte," by Julius Marcusson, pp. 145–150, Verlag von Wilhelm Engelmann, Leipzig (1921).

16 [p. 1025] "A Laboratory Service Test for Pavement Materials," by A. T. Goldbeck, J. E. Gray and L. L. Ludlow, Jr., *Proc. A.S.T.M.*, 34, II, 608 (1934).

17 [p. 1025] "Tentative Method of Test for Thickness of Solid Electrical Insulation" (D 374–36T), A.S.T.M. Tentative Standards 1936, 1011.

18 [pp. 1029, 1033, 1034, 1036 and 1037]. "Standard Methods of Testing Molded Materials Used for Electrical Insulation" (D 48–33), A.S.T.M. Standards 1936, II, 1192; American Standard A.S.A. No: C 59.1–1935, American Standards Association; "Investigation of the Physical Properties of Asphaltic Mixtures at Low Temperatures," by L. F. Rader, *Proc. A.S.T.M.*, 35, II, 559 (1935).

19 [p. 1029] "Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation" (D 229–36T), A.S.T.M. Tentative Standards 1936, 990.

20 [p. 1038] **U. S. Pat.** 1184837 of May 30, 1916 to H. R. Edgecomb.

21 [p. 1039] "Tentative Methods of Testing Electrical Insulating Materials for Resistance to Impact" (D 256–34T), A.S.T.M. Tentative Standards 1936, 905.

22 [p. 1044] "Standard Methods of Testing Molded Materials Used for Electrical Insulation" (D 48–33), A.S.T.M. Standards 1936, II, 1192; American Standard A.S.A. No: C-59.1–1935, American Standards Association; "Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength" (D 149–36T), A.S.T.M. Tentative Standards 1936, 892.

23 [p. 1044] "Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation" (D 176–36T), A.S.T.M. Tentative Standards 1936, 871.

24 [p. 1044] "Standard Methods of Testing Electrical Insulating Oils" (D 117–36), A.S.T.M. Standards 1936, II, 1167.

25 [p. 1044] "Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant" (D 150–36T), A.S.T.M. Tentative Standards 1936, 951.

26 [p. 1044] "Standard Method of Test for Reisitivity of Insulating Materials" (D 257–33), A.S.T.M. Standards 1936, II, 1203; American Standard A.S.A. No: C–59.3–1935.

27 [p. 1045] "Microanalytical Methods in Oil Analysis," by A. H. Gill and H. S. Simms, *J. Ind. Eng. Chem.*, 13, 547 (1921); "Detecting Small Quantities of Petroleum," by E. T. Erickson, *Eng. Mining J.*, 112, 59 (1921); "Methods of Detecting Small Quantities of Petroleum," by K. C. Heald, Bull. Am. Assoc. Petroleum Geol., 5, 620 (1921); "Testing of Road Materials; Using Small Quantities," by Roman Grengg, *Mikrochem.*, 8, 281 (1930).

28 [p. 1045] "Standard Method of Testing Bituminous Mastics, Grouts and Like Mixtures" (D 147–27), A.S.T.M. Standards 1936, II, 1143.

29 [p. 1049] "Determination of the Percentage of Bitumen in Asphaltic Materials," by D. M. Wilson, *Chemistry & Industry*, 50, 599 (1931).

30 [p. 1050] "Extracting Asphalt from Roadbed Mixtures Without Changing its Physical Properties," by Hermann Suida, R. Benigni and W. Janisch, *Asphalt u. Teer*, 31, 197 (1931).

31 [p. 1050] "Laboratory Manual of Bituminous Materials," by Prévost Hubbard, 1st Edition, p. 108, John Wiley & Sons, Inc., New York (1916).

32 [p. 1053] "The Extraction of Bitumens from Mineral Aggregate," by M. R. Walczak and H. I. Rice, *J. Ind. Eng. Chem.*, 12, 738 (1920).

33 [p. 1054] "Separation of Free Carbon from Mineral Matter in the Analysis of Tar Coatings and Tar-Mineral Mixtures," by Ernst Ohse, *Asphalt u. Teer Strassenbautech.*, 33, 441 (1933).

34 [p. 1054] "Determination of Tar Content in Tarred Slags and Stones," by F. J. Nellensteyn, *Asphalt u. Teer*, 30, 543 (1930).

35 [p. 1055] "Standard Methods of Chemical Analysis of Rubber Products" (D 297–36); A.S.T.M. Standards 1936, II, 1249.

36 [p. 1056] "Standard Specifications for Sieves for Testing Purposes" (E 11–26), A.S.T.M. Standards 1936, II, 1413; "Standard Specifications for Round-Hole Screens for Testing Purposes" (E 17–36), A.S.T.M. Standards 1936, II, 1411; "Tentative Definition of the Term Screen (Sieve)" (E 13–28T), A.S.T.M. Tentative Standards 1936, 1329; "Federal Spec. for Sieves; Standard, Testing," RR-S-366, Federal Standard Stock Catalogue, Section IV (Part 5), (Jun. 28, 1932); "Standard Method of Test for Sieve Analysis of Aggregates for Concrete" (C 41–36), A.S.T.M. Standards 1936, II, 353.

37 [p. 1056] "A New Device for the Mechanical Analysis of Concrete Aggregates," by C. N. Forrest, *Proc. A.S.T.M.*, 6, 458 (1906).

38 [p. 1056] "Tentative Method of Test for Determination of Amount of Material Finer than No. 200 Sieve in Aggregates" (C 117–35T), A.S.T.M. Tentative Standards 1936, 488.

39 [p. 1056] "Standard Method of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete" (D 7–27), A.S.T.M. Standards 1936, II, 1077.

40 [p. 1056] "Standard Method of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete" (D 19–16), A.S.T.M. Standards 1936, II, 1076.

41 [p. 1056] "Standard Method of Mechanical Analysis of Broken Stone or Broken

Slag, Except Aggregates Used in Cement Concrete" (D 18–16), A.S.T.M. Standards 1936, II, 1075; "Standard Form of Specifications for Certain Commerical Grades of Broken Stone" (D 35–18), A.S.T.M. Standards 1936, II, 1113.

42 [p. 1056] "Tentative Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter" (D 313–35T), A.S.T.M. Tentative Standards 1936, 850.

43 [p. 1058] "Standard Method of Test for Quantity of Clay and Silt in Gravel for Highway Construction" (D 72–21), A.S.T.M. Standards 1936, II, 1050.

44 [p. 1059] "Standard Method of Decantation Test for Sand and Other Fine Aggregates" (D 136–28), A.S.T.M. Standards 1936, II, 1051.

45 [p. 1060] "Separation and Size Distribution of Microscopic Particles," by P. S. Roller, Tech. Paper No. 490, Bureau of Mines, U. S. Dept. of Commerce, Wash., D. C. (1931); "Some of the Fundamental Physical Characteristics of Mineral Filler Intended for Asphalt Paving Mixtures," by J. S. Miller and R. N. Traxler, *Proc, Assoc. Asphalt Paving Technologists*, p. 53, Detroit, Mich. (Jan. 14, 1932); "Tentative Method of Analysis for the Particle Size Distribution of Sub-Sieve Size Particulate Substances" (E 20–33T), A.S.T.M. Tentative Standards 1936, 1293.

46 [p. 1060] "Value of a Direct Measurement Method for Particle-size Determination," by Henry Green, *J. Industrial Hygiene*, **7**, 155 (1925).

47 [p. 1064] "Microscopic Measurements for the Determination of Particle Size of Pigments and Powders," by E. J. Dunn, Jr., *J. Ind. Eng. Chem.*, Analytical Ed., **2**, 15 (1930).

48 [p. 1064] "Microchemistry of White Pigments and Inerts as they Occur Mixed in Paints," by Henry Green, *J. Ind. Eng. Chem.*, **16**, 667 (1924).

49 [p. 1064] W. M. Weigel, Technical Paper No. 296, U. S. Bureau of Mines, Wash., D. C. (1922).

50 [p. 1064] "Photomicrographic Method for the Determination of Particle Size of Paint and Rubber Pigments," by Henry Green, *J. Franklin Inst.*, **192**, 637 (1921).

51 [p. 1064] "Researches on the Theory of Fine Grinding," by G. Martin, C. E. Blyth and Harold Tongue, Brit. Portland Cement Research Assoc., Pamphlet No. 4 (1924); *Trans. Ceramic Soc.* (England), **23**, 61 (1924).

52 [p. 1066] "A Rapid Method of Determining the Specific Gravity of Pigments and Powders," by E. J. Dunn, Jr., *Proc. A.S.T.M.*, **32**, II, 327 (1932); "Standard Method of Test for Specific Gravity of Pigments" (D 153–27), A.S.T.M. Standards 1936, II, 638.

53 [p. 1066] "Tentative Method of Test for Specific Gravity and Absorption of Fine Aggregate" (C 128–36T), A.S.T.M. Tentative Standards 1936, 514.

54 [p. 1068] "Tentative Method of Test for Specific Gravity and Absorption of Coarse Aggregate" (D 127–36T), A.S.T.M. Tentative Standards 1936, 511.

55 [p. 1070] "Standard Method of Test for Determination of Voids in Fine Aggregate for Concrete" (C 30–22), A.S.T.M. Standards 1936, II, 360; American Tentative Standard A.S.A. No: A 19–1923 American Standards Association.

56 [p. 1070] "Standard Method of Test for Approximate Percentage of Voids in Inundated Fine Aggregate" (C 69–30), A.S.T.M. Standards 1936, II, 361.

* *

CHAPTER XXXIV

1 [p. 1072] "Analysis and Testing of Prepared Roofing," by Herbert Abraham, *J. Ind. Eng. Chem.*, **9**, 1048 (1917).

2 [p. 1072] "Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing" (D 146–27), A.S.T.M. Standards 1936, II, 1153.

3 [pp. 1075 and 1076]. "Tentative Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc, or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules" (D 228–33T), A.S.T.M. Tentative Standards 1936, 852.

4 [p. 1078] "A Supplementary Study of Commercial Instruments for Determining the

Tearing Strength of Paper," by P. L. Houston, *Paper Trade J.*, **50**, 43 (1922); **U. S. Pats.** 1423841 and 1423842 of Jul. 25, 1922 to Armin Elmendorf.

5 [p. 1080] "Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing" (D 146–27), A.S.T.M. Standards 1936, II, 1153.

6 [p. 1082] "Machine for the Determination of the Pliability of Prepared Roofing and the Breaking-point of Bitumens," by C. S. Reeve and F. W. Yeager, *Proc. A.S.T.M.*, **25**, II, 385 (1925).

7 [p. 1086] "Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength" (D 149–36T), A.S.T.M. Tentative Standards 1936, 892.

8 [p. 1086] "Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation" (D 229–36T), A.S.T.M. Tentative Standards 1936, 986.

9 [p. 1086] "Tentative Specifications and Tests for Friction Tape for General Use for Electrical Purposes" (D 69–36T), A.S.T.M. Tentative Standards 1936, 1048; "Tentative Methods of Testing and Tolerances for Woven Tapes" (D 259–36T), A.S.T.M. Tentative Standards 1936, 1206.

10 [p. 1086] "Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation" (D 295–36T), A.S.T.M. Tentative Standards 1936, 1016.

11 [p. 1088] "Zur Bestimmung des Gewichtes der Rohpappeneinlage und des Gehaltes an Tränkmasse in gesandeten Teerdachpappen," by A. Braeutigam, *Teer u. Bitumen*, **30**, 57 (1932); "Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing" (D 146–27), A.S.T.M. Standards 1936, II, 1153.

12 [p. 1091] "Analysis and Testing of Prepared Roofings," by Herbert Abraham, *J. Ind. Eng. Chem.*, **9**, 1048 (1917).

13 [p. 1092] "A Comparison of Abrasion Test Methods for Embedding of Granular Mineral Surfacing on Asphalt Roofing," by H. W. Greider and G. A. Fasold (Appendix I), *Proc. A.S.T.M.*, **36**, I, 453 (1936).

14 [p. 1099] "Proposed Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles" (Appendix II), *Proc. A.S.T.M.*, **36**, I, 461 (1936); "Proposed Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles" (Appendix III), *Proc. A.S.T.M.*, **36**, I, 464 (1936).

15 [p. 1100] "Tentative Methods of Testing Untreated Paper Used in Electrical Insulation" (D 202–36T), A.S.T.M. Tentative Standards 1936, 929.

16 [p. 1103] "Determination of Water in Wool by Distillation," by C. O. M. Steward, *J. Textile Inst.*, **24**, T98 (1933).

17 [p. 1103] "Federal Spec. for Textiles; Test Methods," Federal Standard Stock Catalog, Section IV (Part 5), CCC-T-191 (May 2, 1933); "Standard General Methods of Testing Woven Textile Fabrics" (D 39–36), A.S.T.M. Standards 1936, II, 1334; American Standard A.S.A. No: L 5-1936 American Standards Association.

18 [p. 1104] "A Constant Temperature and Humidity Room for the Testing of Paper, Textiles, etc.," by F. P. Veitch and E. O. Reed, *J. Ind. Eng. Chem.*, **10**, 38 (1918).

19 [p. 1105] "Standard Specifications for Testing Machines" (D 76–36), A.S.T.M. Standards 1936, II, 1291; "Standard General Methods of Testing Woven Textile Fabrics" (D 39–36), A.S.T.M. Standards 1936, II, 1334; American Standard A.S.A. No: L 5–1936 American Standards Association; "Federal Spec. for Textiles; Test Methods," Federal Standard Stock Catalogue, Section IV (Part 5), CCC-T-191 (May 2, 1933).

20 [p. 1107] "Tentative Methods of Testing Untreated Paper Used in Electrical Insulation" (D 202–36T), A.S.T.M. Tentative Standards 1936, 929.

21 [p. 1109] "Test to Determine the Speed with which Felt will Saturate," by Technical Committee Prepared Roofing Association (Feb. 13, 1923); "The Xylene Text for Roofing Felt," by P. W. Codwise, *Paper Trade J.*, **88**, 66 (1929).

22 [p. 1109] "The Anthracene Oil Absorption Test for Roofing Felt," by Wolfgang Malchow, *Teer*, **24**, 179 (1926); "Again the Anthracene Oil Absorption Test," by A. Braeutigam, *Teer*, **24**, 298 (1926); "The Oil Penetration Test for Roofing Felt," by P. W. Codwise,

Paper Mill, **51**, 132 (1928); *Paper Trade J.*, **86**, 54 (1928); "Prüfung von Rohpappen auf Anthraceneölaufnahme," by Staatlichen Materialprüfungsamt, Berlin-Dahlen, *Teer u. Bitumen*, **27**, 604 (1929).

23 [p. 1110] "Test to Ascertain the Thoroughness with which Felt will Saturate," by Technical Committee Prepared Roofing Association, Chicago (Aug. 29, 1922); "The Kerosene Test for Roofing Felt," by P. W. Codwise, *Paper Trade J.*, **87**, 60 (1928); "Testing Unimpregnated Roofing Felt; Proposed Official Method of the Technical Association of the Pulp and Paper Industry," *Paper Trade J.*, **89**, 213 (1929).

24 [p. 1111] "Determination of the Fiber Composition of Roofing Felts," by R. E. Lofton, *Paper Trade J.*, **84**, 57 (1927); "Standard Methods of Analysis of Roofing Felt for Fiber Composition" (D 272–34), A.S.T.M. Standards 1936, II, 1162; "Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles" (D 276–35T), A.S.T.M. Tentative Standards 1936, 1172; "Mikrochemische Papieruntersuchung," by Alois Herzog, Hirschwaldsche Buchhandlung, Berlin (1935).

25 [p. 1115] "A Test for Wool," by H. Le B. Gray, *J. Ind. Eng. Chem.*, **10**, 683 (1918).

26 [p. 1115] "Erfahrungen bei der Bestimmung des Wollgehaltes von Rohpappen auf chemischen Wege," by R. Korn and B. Schulze, *Teer u. Bitumen*, **29**, 34 (1931).

27 [p. 1115] "A Rapid Method for Distinguishing Bleached Sulfate from Bleached Sulfite," by R. W. Shaffer, *J. Ind. Eng. Chem.* (Analytical Edn.), **5**, 35 (1933).

CHAPTER XXXV

1 [p. 1117] "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," by H. A. Gardner, 5th Edition, Institute of Paint and Varnish Research, Wash., D. C. (Oct., 1930); "Standard Methods of Testing Oleo-Resinous Varnishes" (D 154–28), A.S.T.M. Standards 1936, II, 779; "Tentative Methods of Testing Oleo-Resinous Varnishes (Skinning Test, Alkali Resistance, Acid Number)" (D 154–35T), A.S.T.M. Tentative Standards 1936, 588.

2 [p. 1118] "Standard Specifications and Tests for Soluble Nitrocellulose" (D 301–33), A.S.T.M. Standards 1936, II, 806 (section entitled "Consistency").

3 [pp. 1118, 1120 and 1121]. "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," by H. A. Gardner, 8th Edition, Institute of Paint and Varnish Research, Washington, D. C. (1937).

4 [pp. 1118, 1119, 1121, 1122 and 1123] "Tentative Methods of Testing Varnishes Used for Electrical Insulation" (D 115–36T), A.S.T.M. Tentative Standards 1936, 1028.

5 [p. 1120] "An Application of the Pfund Cryptometer," by J. A. Calbeck and C. P. Olander, *Proc. A.S.T.M.*, **22**, II, 515 (1922).

6 [p. 1120] "An Instrument for Measuring the Hiding Power of Paints," by R. L. Hallett, *Proc. A.S.T.M.*, **20**, II, 426 (1920).

7 [p. 1120] "A Photometric Method for Measuring the Hiding Power of Paints," by H. D. Bruce, Tech. Paper No. 306, U. S. Bureau of Standards, Wash., D. C. (1926).

8 [p. 1120] "Tentative Method of Test for Comparative Hiding Power of Paints," (D 344–32T), A.S.T.M. Tentative Standards 1936, 601; "Tentative Method of Test for Comparative Hiding Power of White Pigments" (D 406–35T), A.S.T.M. Tentative Standards 1936, 578; "Standard Method of Test for Mass Color and Tinting Strength of Dry Color Pigments or Pastes" (D 387–36), A.S.T.M. Standards 1936, II, 651; "Standard Method of Test for Tinting Strength of White Pigments or White Pigment Pastes" (D 332–36), A.S.T.M. Standards 1936, II, 647.

9 [p. 1120] "Standard Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units" (D 307–30), A.S.T.M. Standards 1936, II, 822.

10 [p. 1120] "The Measurement of the Gloss of Paints by the Intersoll Glarimeter," by E. F. Hickson, Am. Paint & Varnish Mnfrs. Assoc., Circ. No. 307 (Apr., 1927).

11 [p. 1121] "Measurement of the Thickness of Varnish Films," by E. A. Stoppel, *Proc. A.S.T.M.*, **23**, I, 285 (1923).

12 [p. 1122] "Rostschutzprüfing," by Richard Grün and Hermann Manecks, *Teer u. Bitumen*, 30, 181 (1932).

13 [p. 1122] "Quantitative Determination of Body and Solvent in Varnish," by A. L. Brown, *Proc. A.S.T.M.*, 14, II, 467 (1914); "Determination of Volatile Thinner in Oil Varnish," by E. W. Boughton, Tech. Paper No. 76, Bureau of Standards, Wash., D. C. (Jun. 21, 1916).

14 [p. 1125] "Standard Method of Test for Steam Distillation of Bituminous Protective Coatings" (D 255–28), A.S.T.M. Standards 1936, II, 1150.

15 [p. 1128] "Some Technical Methods of Testing Miscellaneous Supplies," by P. H. Walker, Bull. No. 109 (Revised), Bureau of Chemistry, U. S. Dept. of Agri., Wash., D. C. (Feb. 28, 1910); "The Solubility of Water in Gasoline and Other Organic Liquids," by C. W. Clifford, *J. Ind. Eng. Chem.*, 13, 631 (1921); "Standard Methods of Routine Analysis of White Linseed Oil Paints" (D 215–35), A.S.T.M. Standards 1936, II, 662; "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," by H. A. Gardner, 8th Edition, Institute of Paint and Varnish Research, Wash., D. C. (1937).

16 [p. 1128] "Determination of Volatile Thinners in Oil Varnishes," by A. de Waele, *Analyst*, 43, 408 (1918).

17 [p. 1128] "Standard Method of Test for Flash Point by Means of the Tag Closed Tester" (D 56–36), A.S.T.M. Standards 1936, II, 907; American Standard A.S.A. No: Z 11.24–1936 American Standards Association; American Petroleum Institute Standard A.P.I. No: 509–36.

18 [p. 1128] "Standard Methods of Sampling and Testing Lacquer Solvents and Diluents" (D 268–33), A.S.T.M. Standards 1936, II, 813; "Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosene and Similar Petroleum Products" (D 86–35), A.S.T.M. Standards 1936 II, 870; American Standard A.S.A. No: Z 11.10–1935 American Standards Association; American Petroleum Institute Standard A.P.I. No: 507–35; "Standard Method of Test for Distillation of Crude Petroleum" (D 285–36), A.S.T.M. Standards 1936, II, 863; American Standard A.S.A. No: Z 11.32–1936, American Standards Association; American Petroleum Institute Standard A.P.I. No: 525–36; "Tentative Specifications for Petroleum Spirits (Mineral Spirits)" (D 235–34T), A.S.T.M. Tentative Standards 1936, 581.

19 [p. 1128] "Solvents, Oils, Gums, Waxes and Allied Substances," by F. S. Hyde, D. Van Nostrand Co., New York (1913); "The Examination of Acetone Oils," by W. J. Jones, *J. Soc. Chem. Ind.*, 38, 108T (1918); "Die Lösungsmittel der Fette, Oele, Wachse und Harze," by Hans Wolff-Berlin, Vol. I, Wissenschaftliche Verlagsgesellschaft, m.b.H. Stuttgart (1922); 2nd Edition (1927); "Volatile Solvents and Thinners," by Nöel Heaton, D. Van Nostrand Co., New York (1926); "Lösungsmittel und Hilfsstoffe," by Hiag (1929); "Lösungsmittel und Weichmachungsmittel," by I.-G. Farbenindustrie, A.-G. (1930); "Solvents," by T. H. Durrans, Monograph on Applied Chemistry, Vol. IV, Chapman & Hall, London, 1st Edition (1930); 2nd Edition (1931); 3rd Edition Revised (1933); "Anwendung organischer Lösungsmittel," by E. J. Fischer, Laboratorium Bücher Vol. 30, Verlag von Wilhelm Knapp, Halle (Saale), (1932); "Chemische Technologie der Lösungsmittel," by Otto Jordan, Verlag von Julius Springer, Berlin (1932); "Lösungsmittel und Weichmachungsmittel," by T. H. Durrans and Otto Merz, Verlag von Wilhelm Knapp, Halle (Saale), (1933); "Organic Solvents; Physical Constants and Methods of Purification," by A. Weissberger and E. Proskauer (Translated by R. G. A. New), Clarendon Press, London, H. Milford, Oxford Univ. Press, Oxford (1935); "Decknamen und chemische Zusammensetzung der wichtigsten Lösungsmittel und Weichmacher," by Hans Ulrich, O. Elsner, Berlin (1936).

20 [p. 1128] "Note on the Analysis of Mixed Paints," by C. C. Daubney and B. A. Ellis, *Chem. & Ind.*, 47, 1147 (1928).

21 [p. 1129] "Standard Methods of Routine Analysis of White Pigments" (D 34–33), A.S.T.M. Standards 1936, II, 685; American Standard A.S.A. No: K 15–1933 American Standards Association; "Standard Methods of Routine Analysis of Titanium Pigments" (D 186–33), A.S.T.M. Standards 1936, II, 656; "Standard Methods of Routine Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments and Chrome

Green" (D 126–36), A.S.T.M. Standards 1936, II, 697; "Standard Methods of Routine Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganesese" (D 50–36), A.S.T.M. Standards 1936, II, 710; "Standard Method of Routine Analysis of Dry Cuprous Oxide" (D 283–36), A.S.T.M. Standards 1936, II, 624; "Standard Method of Routine Analysis of Dry Red Lead" (D 49–35), A.S.T.M. Standards 1936, II, 633; American Standard A.S.A. No: K 16.1–1935, American Standards Association; "Standard Method of Routine Analysis of Dry Mercuric Oxide" (D 284–33), A.S.T.M. Standards 1936, II, 629.

22 [p. 1129] "Standard Methods of Test for Coarse Particles in Dry Pigments and Coarse Particles and Skins in Mixtures of Pigments and Vehicles" (D 185–29), A.S.T.M. Standards 1936, II, 621.

23 [p. 1130] "Colorimetric Determination of Cobalt in Varnishes," by E. G. Jones, *J. Soc. Chem. Ind.*, **37**, 630A (1918); "Detection of Lead, Manganese and Cobalt in Varnishes and Oil Lacquers," by H. Vollmann, *Farben. Z.*, **27**, 1943 (1922).

24 [p. 1130] "The Determination of Rosin in Varnishes," by A. H. Gill, *J. Am. Chem. Soc.*, **28**, 1723 (1906); "Detection of Resin in Dryer," by E. W. Boughton, Tech. Paper No. 66, Bureau of Standards, Wash., D. C. (Jan. 15, 1916); "Determination of Rosin in Rosin-Bitumen Mixtures," by A. Wogrinz and P. Vari, *Chem. Zeit.*, **43**, 506 (1919); "Methods of Varnish Analysis," by W. T. Pearce, *J. Ind. Eng. Chem.*, **11**, 200 (1919).

25 [p. 1130] "Determination of Oil and Resin in Varnish," by E. W. Boughton, Tech. Paper No. 65, Bureau of Standards, Wash., D. C. (Feb. 19, 1916).

CHAPTER XXXVI

1 [p. 1132] "Merkblatt über die Verwendung von Strassenbaubefestigungen," by Forschungsgesellschaft für das Strassenwesens, e.V., Nov. 1, 1935.

2 [p. 1132] "Methode zur Bestimmung der Brechbarkeit von Bitumenemulsionen," by A. Caroselli, *Bitumen*, **6**, 61 (1936).

3 [pp. 1133, 1139 and 1140] "Modern Paving Emulsions, Types, Characteristics and Test Methods," by C. L. McKesson, *Proc. A.S.T.M.*, **31**, II, 841 (1931); "Tests for Modern Paving Emulsions," by C. L. McKesson, *Can. Engr.*, **61**, 15 (1931); "Tentative Methods of Testing Emulsified Asphalts" (D 244–36T), A.S.T.M. Tentative Standards 1936, 744; American Association State Highway Officials Standard A.A.S.H.O. No: T–59.

4 [p. 1135] "A Study Concerning a Modification of the Present Miscibility Test and its Relationship with Probable Settlement Results," by C. A. Downing, *Proc. A.S.T.M.*, **35**, II, 546 (1935).

5 [p. 1136] "Myers' Demulsification Test for Bituminous Emulsions," by P. R. Smith, *J. Ind. Eng. Chem.* (Analytical Edition), **4**, 105 (1932).

6 [p. 1137] "Chemistry and Physics of Street Tar," by Heinrich Mallison, *Gas- u. Wasserfach.*, **72**, 1215 (1929); "Stabilization of Aqueous Dispersions of Asphalt," by A. W. Hixson and J. M. Fain, *J. Ind. Eng. Chem.*, **24**, 1336 (1932).

7 [p. 1138] "Modern Paving Emulsions, Types, Characteristics and Test Methods," by C. L. McKesson, *Proc. A.S.T.M.*, **31**, II, 841 (1931).

8 [p. 1139] "Asphaltic Emulsions for Economical Highways," by C. L. McKesson, *Civil Eng.*, **1**, 1255 (1931).

9 [p. 1143] "Water Determination in Bitumen Emulsions," by G. Keppler and Kurt Dohse, *Asphalt u. Teer Strassenbautech.*, **32**, 722 (1932).

10 [p. 1145] "Separation of the Original Tar from Road Tar Emulsions," by H. Wagner, *Mitt. Materialprüf*, p. 220 (1932).

11 [p. 1145] "Bituminous Emulsions," by Julius Marcusson, *Mitt. königl. Materialprüfungsamt*, **36**, 279 (1918); "Analysis of Cold Asphalts (Emulsions)," by Julius Marcusson, *Mitt. deut. Materialprüf.*, Spec. No. 6, pp. 87–88 (1929); *Asphalt u. Teer Strassenbautech.*, **29**, 510 (1929).

CHAPTER XXXVII

1 [p. 1147] "Geschichte der Photographie," by Elder, I, 153 (1905).

2 [p. 1147] "Syrian Asphaltum for Printing Plates," by C. Fleck, *J. Soc. Chem. Ind.*, **23**, 268 (1904); "Experimental Contribution to the Knowledge of the Bitumens," by G. Maderna, *J. Soc. Chem. Ind.*, **28**, 694 (1909); "Light Sensibility of Asphalt," by Jacques Errera, *Trans. Faraday Soc.*, **19**, 314 (1923).

3 [p. 1147] "A New Method to Increase the Light-Sensitivity of Asphalts," by E. Valenta, *Chem. Zentr.*, **20**, 685 (1891); "Increase of Sensitiveness of Asphalt," by E. Valenta, *Phot. Korr.*, **47**, 236 (1910); "Sensitiveness of Asphalt to Light," by A. Rosinger, *Chem. Ztg.*, **36**, 243 (1912); *Kolloid Zts.*, **15**, 177 (1914); "Chemistry of Asphalt and Especially Photo-chemical Properties," by Paul Gödrich, *Monatshefte für Chem.*, **36**, 535 (1915); "Observations on the Action of Sulfur Monochloride on Bituminous and Tarry Substances," by J. V. Meigs, *J. Ind. Eng. Chem.*, **9**, 655 (1917); "The Products Obtained by the Dry-distillation of Asphalt from Matita," by C. Nicolescu-Otin, *Bull. Sec. Sci. Acad. Roumaine*, **5**, 129 (1920); "Increasing the Optically Active Constituents of Bitumens by Means of Ozone," by Jenö Tausz, *Chem. Ztg.*, **43**, 225 (1919).

4 [p. 1148] "The Light-sensitiveness of Petroleum Asphalt," by Paul Gödrich, *Chem. Ztg.*, **39**, 832 (1915); *Monatshefte für Chem.*, **36**, 535 (1915).

5 [p. 1148] **U. S. Pat.** 940808 of Nov. 23, 1909 to John Hartnell, Jr.; **Ger. Pats.** 211329 of Aug. 24, 1907 to Bertha Trautmann; 270105 of Apr. 3, 1913 to Josef Rieder; 301929 of Sep. 12, 1916 to Josef Rieder; 309376 of Jan. 18, 1918 to Josef Rieder; 436045 of Jul. 18, 1925 to Josef Rieder.

6 [p. 1148] "The Influence of Sunlight on Paints and Varnishes," by Maxmilian Toch, *J. Soc. Chem. Ind.*, **27**, 311 (1908).

7 [p. 1148] "Weathering of Bituminous Coatings," by J. P. Pfeiffer, *Verfkroniek*, **8**, 43 (1935).

8 [p. 1148] "The Effect of Exposure on Bitumens," by Prévost Hubbard and C. S. Reeve, *J. Ind. Eng. Chem.*, **5**, 15 (1913).

9 [p. 1148] "Some Experiments on Technical Bitumens," by S. R. Church and J. M. Weiss, *Proc. A.S.T.M.*, **15**, II, 275 (1915).

10 [p. 1148] "The Effect of Exposure on Tar Products," by C. S. Reeve and B. A. Anderton, *J. Franklin Inst.*, **182**, 463 (1916).

11 [p. 1148] "The Effects of Exposure on Some Fluid Bitumens," by C. S. Reeve and R. H. Lewis, *J. Ind. Eng. Chem.*, **9**, 743 (1917).

12 [p. 1148] "Data on the Oxidation of Automobile Cylinder Oils," by C. E. Waters, Tech. Paper No. 73, U. S. Bureau of Standards, Wash., D. C. (1916).

13 [p. 1149] "Oxygen Absorption Test on Asphalt Constituents," by R. R. Thurston and E. C. Knowles, *J. Ind. Eng. Chem.*, **28**, 88 (1936); "A Study of Road Tars," by R. H. Lewis and J. Y. Welborn, *Public Roads*, **17**, 89 (1936).

14 [p. 1150] "The Testing of Bitumens for Paving Purposes," by A. W. Dow, *Proc. A.S.T.M.*, **3**, 359 (1903).

15 [p. 1150] "Increase in Viscosity of Asphalts with Time," by R. N. Traxler and H. E. Schweyer, *Proc. A.S.T.M.*, **36**, Part II, 544 (1936).

16 [p. 1151] "Action of Water on Asphalt Pavements," Chapter XXIV, p. 460, "The Modern Asphalt Pavement," by Clifford Richardson, 2nd Edition, John Wiley & Sons, New York (1908); "Permeability of Bituminous Paints to Water," by H. Walther, *Asphalt und Teer*, **35**, 207 (1935); *Chem. Zentr.* II, 786 (1935); "Die Wasseraufnahme bituminöser Anstriche," by Heinrich Mallison, Vedag-Buch 1936, p. 144, C. Falian, Berlin (1936).

17 [p. 1151] "Beiträge zur Chemie der Asphalte mit besonderer Berücksichtigung ihrer photochemischen Eigenschaften," by Paul Gödrich, *Monatsh. Chem.*, **36**, 535 (1915); "Ueber die Ursachen des Ausbleichen der Asphaltgesteine," by Edmund Graefe, *Asphalt u. Teer Strassenbautechnik*, **32**, 625 (1932); "Ueber Beschädigungen an Zinkrinnen und Vorstoss

blechen von Bitumenpappdächern," by E. Deiss, Vedag-Buch 1936, p. 123, C. Falian, Berlin (1936).

18 [p. 1151] "A Modified Accelerated Weathering Test for Asphalts and Other Materials," by O. G. Strieter and H. R. Snoke, Research Paper RP-886, National Bureau of Standards, U. S. Dept. of Commerce, Wash., D. C. (May 1936); "Choice and Application of Bituminous Paints," by H. Walther, *Oel, Kohle, Erdöl, Teer*, **12**, 191 (1936).

19 [p. 1152] "A Study of Some Liquid Asphaltic Materials of the Slow-curing Type," by R. H. Lewis and W. O'B. Hillman, *Public Roads*, **15**, 85 (1934); "Further Studies of Liquid Asphaltic Materials," by R. H. Lewis and W. O'B. Hillman, *Public Roads*, **16**, 6 (1935).

20 [p. 1154] **Ger. Pat.** 239289 of Nov. 1, 1908 to Wolfgang Ostwald and Walter Ostwald.

21 [p. 1154] **Ger. Pat.** 249955 of Mar. 23, 1909 to Wolfgang Ostwald and Walter Ostwald.

22 [p. 1154] **Ger. Pat.** 526783 of Feb. 20, 1927 to Ges. für Strahlungschemie, G.m.b.H.

23 [p. 1154] "Metallates," by H. A. Gardner, *J. Ind. Eng. Chem.*, **28**, 1020 (1936).

24 [p. 1154] **Australian Pat.** 2866/31 of Jul. 14, 1932 to C. F. Broadhead and R. S. Andrews.

25 [p. 1155] "The Formation of Blisters on Asphalt Mastic Roofs," *R. I. B. A. J.* **41**, 138 (1933); *Building Sci. Abs.*, No. 282 (1934); "Blisters in Floated Asphalt," P. Marx, *Strassenbau*, **26**, 137 (1935); *Building Sci. Abs.*, **8**, No. 1120 (1935).

26 [p. 1158] "The Durability of Prepared Roll Roofings," by Henry Giese, H. J. Barre and J. B. Davidson, Bull. No. 109, Iowa Engineering Experiment Station, Iowa State College, Ames, Iowa (May 4, 1932); "What Determines the Length of Life of Prepared Roll Roofings?", by Henry Giese, H. J. Barre and J. B. Davidson, Bull. No. 304, Agricultural Experiment Station, Iowa State College, Ames, Iowa (Jun., 1933); "A Study of the Weathering Quality of Roofing Felts Made from Various Fibres," by O. G. Strieter, Research Paper RP-888, National Bureau of Standards, U. S. Dept. of Commerce (May 1936); *J. of Research*, National Bureau of Standards, **16** (May 1936).

27 [p. 1159] "Exposure Tests on Colorless Waterproofing Materials," by D. W. Kessler, Tech. Paper No. 248, Bureau of Standards, Wash., D. C. (Jan. 7, 1924); "A New Test for Predicating the Durability of Varnishes (The Photochemical Embrittling Test)," by J. H. Wilson, Bureau of Standards Research Paper No. 333, Wash., D. C. (1931); "Bituminous Coatings," by G. G. Sward and Francis Scofield, Circular No. 504, National Paint, Varnish and Lacquer Assoc. (Apr. 1936).

28 [p. 1160] "Ueber die Veränderung verschiedener Bitumensorten an der Oberfläche," by J. Oberbach and O. Pauer, *Teer u. Bitumen*, **32**, 411 (1934).

29 [p. 1160] "Liquid Asphaltic Road Materials," by R. H. Lewis and W. O'B. Hillmann, *Public Roads*, **16**, 97 (1935).

30 [pp. 1161 and 1169] "Accelerated Tests of Organic Protective Coatings," by P. H. Walker and E. F. Hickson, *Bureau of Standards J. Research*, **1**, 1 (1928); *J. Ind. Eng. Chem.*, **20**, 591 (1928); "Accelerated Weathering Properties of Oklahoma Asphalts," by P. G. Shelley, Circ. No. 19, Oklahoma Geol. Survey, Norman, Okla. (Oct., 1929); "Accelerated Tests of Asphalts," by O. G. Strieter, Research Paper No. 197, *Bureau of Standards J. Research*, **5**, 247 (1930); "Bituminous Materials Tested," by O. G. Strieter, *Commercial Standards Monthly*, **7**, 151 (1930).

31 [p. 1167] "Proposed Method for Accelerated Weathering Tests on Bituminous Materials," *Proc. A.S.T.M.*, **33**, Part I, 381 (1933).

32 [p. 1171] "A New Test for Predicating the Durability of Varnishes (The Photochemical Embrittling Test)," by J. H. Wilson, *Bureau of Standards J. Research*, **7**, 73 (1931); "Choice and Application of Bituminous Paints," by H. Walther, *Oel u. Kohle*, **12**, 191 (1936).

33 [p. 1171] "The Effect of Mineral Fillers on the Serviceability of Coating Asphalts," by O. G. Strieter, *Proc. A.S.T.M.*, **36**, Part II, 486 (1936).

INDEX

NOTE—*Italicized* words are in foreign use.

Abrasion test for solvent compositions, 1120
Absolute viscosity test, 817, 920
Absorption test of:
  bituminized fabrics, 1084
  bituminized mineral aggregates, 1018, 1029
  bituminous dispersions, 1139
  bituminous-solvent compositions, 1121
  bituminous substances, 785, 1150
  coal-tar pitch, 368, 698
  electrical insulating papers, 709
  molded compositions, 1029
  paving compositions, 1018
A. C. (asphalt cement), 570
Accelerated weathering test, 1161, 1177
Acetone, solubility in, 78, 300, 970, 971, 973
Acetone oils, 292
Acid-asphalt, 432
Acid-coke, 460, 463
Acid-naphthalene oil, 366
Acid-oil distillate, 415, 460
Acid-oils, 294, 415
Acid-proof enamel, 534
  covering powder of, 535
  resistance to acids, 534
Acid-proof mastic, 604, 605
Acid-resisting coatings, 534, 737
Acid-resisting tests, 739, 1122
Acid-resisting varnish, 749
Acids:
  asphaltous, 66, 1008
  free, 1001
  mineral, reactions of, 58, 78, 401
  resin, 1001
  tar, 366, 991
Acid-sludge, 320, 321, 333, 415, 416, 460
Acid-sludge asphalt, 432
Acid-sludge pitch, 321, 323
Acid-tar, 460, 463
Acid value, 457, 1001
Acoustical blocks, 719
Acoustical compositions, 513
Acoustical felts, 703, 714
Adhesion (adhesiveness), 855
Adhesion test of bituminous-solvent compositions, 1120
Adhesion test of tape, 1087
Adhesive compositions for:
  built-up roofs, 491, 493, 526, 628
  damp-proofing, 526, 527
  waterproofing, 368, 491, 526, 527, 697
Adhesive or adhesiveness test, 855
  Brown's method, 856
  Riehm's method, 856
  Wedmore's method, 856
Adhesivemeter, 856
Adhesiveness:
  decreasing of, 489
  increasing of, 483, 527
Adsorption of bituminous substances, 525
Adsorptive capacity of mineral fillers, 1066
Aegerite, 264
Aeonite, 264
Africa:
  albertite in, 270
  asphalts in, 226
Aged surface of bituminous substances, 784
Aggregates, see "bituminized discrete aggregates", also "mineral aggregate"
Aging index, 1150
Agitator, 504, 506
Agriculture, early use of asphalt in, 38
Air-drying paints, 740
Air-drying varnish, 740
Air-separation test, 1060
Alabama, asphalt in, 168
Albania, asphalt in, 29, 31, 33, 36, 151, 206
Alberta:
  asphalt in, 77, 123, 177
    blowing of, 179
    extraction with water, 123, 178
  glance pitch in, 206
Albert coal, 48, 267
Albert shale, 331
Albertite, 266
  characterization of, 266
  classification of, 55, 57, 62
  depolymerization of, 72, 473
  discovery of, 48, 267
  metamorphosis of, 95, 262
  properties of, 262, 267, 778
Albertite in:
  Africa, 270
  Australia, 270
  Canada, 48, 267

Albertite in—*Continued:*
Europe, 269
Falkland Islands, 269
Germany, 269
North America, 267
Nova Scotia, 48
Portuguese West Africa, 270
South America, 269
United States, 269
Utah, 269
Albertite shales, 276
Albino asphalt, 631
Alcohol, solubility in, 78, 726
Algeria, asphalt in, 226
Aliphatic hydrocarbons, 281, 728
Alkali-resisting test, 1122
Alkalies, reactions of, 77, 78, 1000
Alsace-Lorraine, asphalt in, 124, 196
extraction with water, 124
Alternating-stress viscosity test, 825, 845
Aluminium foil, use for:
felts, 654, 714
paints, 740
papers, 710, 713
pipe-wrapping, 539
Amber tar, 280
Amber-tar pitch, 280
Amicrons, 803
Amosite asbestos, 624, 745
Ampelitis, 2, 34, 37
Analysis of:
bituminized fabrics, 1072
bituminized discrete aggregates, 1044
bituminous dispersions, 1140
bituminous-solvent compositions, 1117
dried paint films, 1131
expansion joints, 1072
floor coverings, 1072
insulating tape, 1072
papers, 1072
roofings, 1072, 1090
saturated felts, 1072, 1088
shingles, 1072
Analysis, ultimate, 68, 982
Analytical-balance method of specific gravity, 799
Anhydrides, 1002
asphaltous-acid, 66, 1008, 1010
Aniline, solubility in, 966
Animal fats and oils, 479, 482, 722, 723
Animal-oil tar, 280
Animal-oil-tar pitch, 280
Animal theories of petroleum origin, 92
Anthracene, 283, 346, 366, 381
Anthracene oil, 346, 356, 364, 366
Anthracene-oil tar, 280
Anthracene-oil-tar pitch, 281, 376
Anthracene pitch, 58, 376
hardening of, 376
sulfurized, 376
Anthracite coal, 97
classification of, 55, 57, 62
metamorphosis of, 94
properties of, 778
Anthracoxenite, 96
Anthraquinone reaction, 1015
Anti-corrosive paints, 741
Anti-fouling paints, 740, 741
Anti-oxidants, effect of, 722, 1128, 1154
Anti-oxygens, 1154
Anti-solvent, 126
Apparent specific-gravity, 1070
Arabia, asphalt in, 33, 220
Arcadian shale, 331
Archangel tar, 297
Argentina:
asphalt in, 190
grahamite in, 259
Argulite, 173
Arkansas:
asphalt in, 167
impsonite in, 271
Arkosite, 271
Armature carbons, 546
Armature windings, 542
Armored bituminized fabrics, 615
Aromatic hydrocarbons, 281, 283, 237, 381, 728
Artificial asphalts, see "petroleum asphalts"
Artificial lumber, 557
Asbestos, varieties of, 624, 745
Asbestos-cement panels, 546
Asbestos-felt, 624, 642, 646, 653, 660
production of, 110
Asia:
asphalt in, 152, 216
elaterite in, 263
gilsonite in, 236
ozokerite in, 134
Asia Minor, asphalt in, 220
Asiatic Russia:
asphalt in, 152, 220
elaterite in, 263
Asphalt:
acid, 432
acid-sludge, 432
aging index of, 1150
agitation of, 117
albino, 631
associated minerals, 83
associated with mineral matter, 155
atomizing, 565
behavior under heat, 73
Bermudez, 50
blending of, 476, 483, 491
blown, see "blown petroleum asphalt"
briquetting of, 104
Buton (Boeton), 224
calendering of, 551, 555

Asphalt—*Continued:*
cellular, 613
chemistry of, 46
Chemoasphalte, 58
chlorinated, 80, 444, 453, 628, 721, 752
classification of, 55, 62
coefficient of expansion of, 872, 874, 875
colloidal carbon in, 66
colloidal nature of, 66, 70, 93, 186
comminution of, 121, 158, 169, 201, 565
composition of, 54
condensed, 431
cracking of, 424, 428, 438, 453
cracking-coil, 438, 439
cut-back, 104, 414, 431, 571
definition of, 56
dehydration of, 114
depolymerization of, 72
Derna, 204
dielectric strength of, 542
diffusibility of, 788
discovery in Cuba, 41
discovery in Trinidad, 40
dispersions of, 496, 503
distillation of, 39, 40, 118, 119, 203, 204, 206, 209, 280
distinguishing from coal-tar pitch, 524, 997
distinguishing from fatty-acid pitch, 403
distinguishing from lignite-tar pitch, 323
Dubb's, 431, 452
early uses in:
agriculture, 38
asphalt-block pavements, 22
Biblical times, 5, 19, 42
boats, 5, 14, 19, 20, 28, 31
bridges, 24
calking ships, 40, 42, 151, 191, 219
cementing objects, 1, 8
charms, 39
chewing-gum, 49
distillation, 39, 40
embalming, 17, 33
embankments, 26, 27
floors, 8, 24
fuel, 33
Greek fire, 30, 39, 40
husbandry, 38
lighting, 31
love-potions, 39
mastic, 8, 15, 16, 20, 22
medicine, 18, 30, 35, 37, 38, 39
mortar, 8, 12, 20, 21, 22, 23, 25, 26, 28, 29, 31, 33, 37, 151
mummies, 17, 18, 32, 37
ointments, 36, 44
ornaments, 5, 7, 11, 12, 13
paints, 16, 21, 31, 35, 41
pavements, 22
plumbing, 24
punishment, 26
rings, 5
sculpture, 12, 13, 14
ships, 40, 41, 42, 151, 191, 219
stair-treads, 8
statues, 10, 11, 13, 16, 19
tablets, 14
tree-surgery, 38
tunnels, 21, 31
warfare, 29, 30, 35, 38, 29, 40
waterproofing, 8, 10, 21, 23, 31, 35
effect of anti-oxidants on, 722, 1128, 1154
effect of blowing on, 446
effect of heat on, 72, 89, 492
effect of light on, 1011, 1147
electrical resistance of, 542, 543
emulsified, 104
extraction of, 122, 125, 165, 204
by means of solvents, 125
by means of water, 122, 124, 165, 177, 178
extrusion of, 551, 554
fillers for, 494
filling veins, 86
formation of, 73
geology of, 82
heat content of, 873
heating of, 115
hydrogenation of, 75
impregnated rock, 86
interfacial tension of, 863
iodine number of, 80
lake, 93, 182
land, 189
lead in, 462
Limmer, 43, 197, 593, 604
liquid, 138, 431
Lobsann, 196
Maracaibo, 149
mastic, 104, 604
metamorphosis of, 93, 94
mineral constituents in, 70
mining methods, 112
mixtures with:
coal-tar pitch, 477, 484, 1154
chlorinated rubber, 486, 541
latex, 487
rubber, 486, 541, 549
wax, 480
molding of, 550
molybdenum in, 71
native, see "native asphalt"
Neuchatel, 43, 195
nickel in, 16, 71
oily medium of, 66, 67
origin of, 44, 54
origin of word, 1
overheating of, 492

Asphalt—*Continued:*
oxidized, or oxygenized, or oxygenated, or oxidized petroleum, see "blown petroleum asphalt"
oxygenation of, 74
paraffin wax in, 630
paving, 104
petroleum, 409, 431
polymerization of, 72, 455, 714, 1150
production of, 98
properties of, 54, 478, 479, 778
pumping of, 119
pure, 139
pyrogenous, 56, 63, 409, 478, 778
Ragusa, 209, 280, 593, 604, 609
reactions with:
acids, 77, 78, 401
alkalies, 58, 79, 1000
chlorine, 58, 80, 444, 453
formaldehyde, etc., 59, 79, 301, 376, 377, 401, 486, 999
furfurol, 59
gases, 74, 444
halogens, 58, 80, 444, 453
hydrogen, 58, 74, 486
iodine, 58, 80
liquid sulfur dioxide, 77
metallic salts, 58, 81, 497, 808
metalloids, 79
mineral acids, 58
nitric acid, 78, 401, 486, 739
oxygen, 58, 74, 444
ozone, 58, 301, 444, 489, 755
phosphoric acid, 58, 79, 497, 509
phosphorus, 58, 80
phosphorus derivatives, 58
precipitation agents, 58
selenium, 80
sulfides, 453
sulfur, 58, 79, 453, 486
sulfur derivatives, 58, 453
sulfur dichloride, 79, 383, 453, 549
sulfur dioxide, 77
sulfuric acid, 78, 401, 453, 460, 486
sulfuric acid and formaldehyde, 79, 999
sulfur trioxide, 78
refined, 570
refining of, 37, 114
residual, see "residual asphalt"
rock, see "rock asphalt"
roofing, 104
sampling of, 757
sawing into blocks, 209, 211
sedimentation of, 122
seepages of, 84
Sélénitza, 29, 31, 33, 36, 151, 206
semi-liquid, 138
Seyssel, 43, 45, 46, 192, 604
shipping of, 113
Sicilian, 29, 33, 36, 208, 593
sludge, see "sludge asphalt"
solubility of, 54, 729
specific heat of, 872
sponge, 565, 613, 1154
spraying of, 128
storage of, 128
straight-run, 414
sulfurized, 58, 431, 452, 486
surface-tension of, 484, 521, 860
sweating of, 784
synthetic, 784
Tataros, 24, 204
thermal expansion of, 874
transportation of, 113, 148, 185, 208
Trinidad, 40, 67, 70, 81, 93, 182
ultra-microscopic examination of, 524, 802
uranium in, 71
use by:
Assyrians, 25
Babylonians, 20
Egyptians, 16
Incas, 41
Indians, 15
Lake Dwellers, 27
Sumerians, 5
use in Biblical times, 5, 19, 42
Val de Travers, 43, 48, 195, 593, 604
vacuum distillation of, 454
vanadium in, 16, 71, 259, 260, 273
veins of, 86
voids in, 801
waterproofing, 104
weather-resistance of, 477, 478
word in:
Accadian, 1
Arabic, 219
French, 1, 59
German, 59
Greek, 1, 59
Latin, 1
Rumanian, 431
Russian, 59, 215, 431
Spanish, 59
Tartar, 431
Turkish, 219
wurtzilite, see "wurtzilite asphalt"
Asphalt-aging index, 1150
Asphalt-bearing shales, 275
Asphalt binders, 491, 567, 570 (insert), 571
Asphalt-block pavements, 570 (insert), 595
early use of, 22
Asphalt bottoms, 422
Asphalt brick-siding, 681
production of, 109
Asphalt bridge-planking, 616
Asphalt broken-stone pavements, 583
Asphalt built-up roofs, 686

Asphalt cement, 491, 570
Asphalt-concrete pavements, 570 (insert), 571
Asphalt cooler, 413
Asphalt-cork compositions, 552
Asphalt dust, 565
  for coating pipes, 565
  for explosives, 565
  for plastics, 565
Asphalte, 1, 56, 604
Asphalte comprimé, 589
Asphalte coulé, 604
Asphaltenes, 47, 66, 73, 74, 167, 964, 1008, 1011
  behavior of, 73
  molecular weight of, 1010
  nitrated, 1011
  removal of, 470
  synthesis of, 74
Asphalt-fibrous roof-coatings, 737, 741
Asphalt filaments, 565
Asphalt fillers for pavements, 491, 570 (insert), 594, 603
Asphalt floor-covering, 693
Asphalt flux, 428
Asphalt grout, 531, 570 (insert)
Asphaltic carboids, 1008
Asphaltic cement, 491, 493
Asphaltic coal, 47
Asphaltic-concrete pavements, 570 (insert), 571
Asphaltic constituents, test for, 1007
  effect of blowing on, 1010
Asphaltic fillers, 574 (insert), 595, 603
Asphaltic limestone, 56, 62
Asphaltic paving binders, 570 (insert), 571
Asphaltic petroleum, 55, 62, 73, 409, 410, 778
  classification of, 55
Asphaltic pyrobitumen, 262
  behavior under heat, 73
  classification of, 55, 57, 62
  composition of, 54
  copper in, 272
  cracking of, 72
  definition of, 57
  depolymerization of, 72
  differentiation of, 262
  effect of heat on, 72, 73
  hydrogenation of, 486
  metamorphosis of, 89, 93
  origin of, 54, 89
  production of, 98, 100, 102
  properties of, 54, 778
  solubility of, 54
  vanadium in, 273
  veins of, 86
Asphaltic pyrobituminous shales, 276
  classification of, 57, 62
Asphaltic pyrobituminous shales—*Continued:*
  differentiation of, 262
  fluxing with coal-tar pitch, 485
  metamorphosis of, 94, 95
  properties of, 778
Asphaltic resins, 66, 167, 470, 1008, 1011
  fluxing of, 471
  from pressure-tars, 471
  nitrated, 1011
  properties of, 470
  recovery of, 470, 471
  sulfurized, 1012
  use in bituminized fabrics, 471
  use in bituminous dispersions, 471
  use in bituminous-solvent mixtures, 471
Asphaltic saturants, 626
Asphalt in:
  Africa, 226
  Alabama, 168
  Albania, 29, 31, 33, 36, 151, 206
  Alberta, 123, 177
  Algeria, 226
  Alsace-Lorraine, 124, 196
  Arabia, 33, 220
  Argentina, 190
  Arkansas, 167
  Asia, 152, 216
  Asia Minor, 220
  Asiatic Russia, 152, 220
  Australia, 223
  Austria, 124, 203
  Babylonia, 5, 6, 30, 31, 32, 33, 41
  Baku, 39, 40
  Brazil, 190
  Buton (Boeton) Island, 224
  California, 141, 174
  Canada, 177
  China, 221
  Colombia, 191
  Cuba, 144, 180
  Czecho-Slovakia, 204
  Dead Sea, 19, 29, 30, 32, 33, 35, 36, 40, 88
  Dutch East Indies, 224
  Ecuador, 191
  Egypt, 221
  Europe, 150, 191
  France, 45, 124, 150, 191
  Germany, 40, 197
  Greece, 28, 151, 212
  Hungary, 204
  India, 16, 37, 221
  Indiana, 159
  Iraq (Irak), 5, 21, 38, 152, 219
  Italy, 124, 206
  Japan, 221
  Jugoslavia, 202
  Kentucky, 139, 155
  Levant States, 152, 216
  Louisiana, 169

Asphalt in—*Continued:*
Madagascar, 226
Manitoba, 180
Mesopotamia, 5, 24, 27, 33, 152, 219
Mexico, 143, 180
Missouri, 159
Nigeria, 226
North America, 139, 155
Oklahoma, 140, 159
Oregon, 143
Persia, 5, 15, 28, 33, 37, 43
Philippines, 153
Portugal, 212
Rhodesia, 226
Rumania, 205
Russia, 124, 151, 214
Sakhalin, 152
Sicily, 29, 33, 36, 208
South America, 146, 182
Spain, 197, 213
Switzerland, 43, 195
Syria, 152, 216
Texas, 169
Trinidad, 40, 67, 70, 81, 93, 182
Turkey-in-Asia, 29, 220
United States, 139, 155
Utah, 140, 172
Venezuela, 50, 146
Wyoming, 173
Asphaltites, 227
behavior under heat, 73
classification of, 55, 62
comminution of, 121
composition of, 54
definition of, 56
differentiation of, 227
distillation of, 230
distinguishing from residual asphalts, 456
effect of heat on, 73
fluxing of, 478, 492
geology of, 82
heating of, 492
hydrogenation of, 486
metamorphosis of, 93, 94, 262
mineral constituents in, 70
mining methods, 112, 233
molybdenum in, 71
nickel in, 71
nitrated, 726, 752
origin of, 54
production of, 98, 100, 102
properties of, 54, 478, 778
sampling of, 771
shipping of, 113
solubility of, 54, 525 (insert), 729
transportation of, 113
uranium in, 71, 240
vanadium in, 71, 240, 259, 260
veins of, 86
Asphaltites—*Continued:*
weather-resistance of, 478
word in:
Arabic, 28
Greek, 2
Latin, 2
Persian, 28, 39
Asphaltites in:
Argentina, 259
Asia, 236
Barbados, 237
Central America, 241
Colombia, 241
Colorado, 252
Cuba, 238, 254
Europe, 242
Germany, 242
Iraq (Irak), 243
Levant States, 242
Mesopotamia, 243
Mexico, 239, 253
Nicaragua, 241
North America, 230, 237, 245
Oklahoma, 247
Oregon, 235
Peru, 260
Russia, 236
Salvador, 241
San Domingo, 238
South America, 241, 256
Syria, 242
Texas, 247
Trinidad, 256
United States, 230, 240, 245
Utah, 227, 230, 240
West Indies, 237, 256
West Virginia, 245
Asphaltites Lake, 30, 36
Asphalt jelly, 566
Asphalt lakes, 84, 146, 152, 182, 190
Asphalt-leather compositions, 557
Asphalt-macadam pavements, 570 (insert), 571, 583
Asphalt mastic, 104, 604, 610
acid-proof, 604, 605
examination of, 1017
Asphalt-mastic block, 605
Asphalt-mastic cake, 201, 605
Asphalt-mastic floors, 604
Asphalt-mastic foot-pavements, 46, 604
Asphalt-mastic roofs, 609
Asphalt medium, 66
Asphalt micelle, 66, 67
Asphalt mortar, 531
Asphaltos, 2, 19
Asphaltous-acid anhydrides, 66, 1008, 1010
Asphaltous acids, 66, 1008
Asphalt paints, 720
early uses of, 16, 21, 31, 35, 41

Asphalt pavements, 709
early uses of, 22, 45, 46, 47, 48, 49, 50
Asphalt paving-blocks, 570 (insert), 571
Asphalt pipes, 713
Asphalt plastic-cement, 744, 746
production of, 109, 110
Asphalt powder, 565
Asphalt primer, 699, 733
Asphalt putty, 688, 744, 746
Asphalt resins, 1008
Asphalt roofs, 617
Asphalt rugs, 695
Asphalt-saturated felt, 634, 642, 693, 697
production of, 109, 110
Asphalt saturator, 635, 645
Asphalt seepages, 84
Asphalt shingles, 658
Asphalt sidings, 681
Asphalt sponge, 565, 1154
Asphalt springs, 24, 26, 84
Asphalt step-treads, 557
Asphalt tar, 280
Asphalt-tar pitch, 281
Asphalt tiles, 201, 598
Asphaltum, see "asphalt"
Asphaltum oil, 431
Asphaltum pot, 44
Asphalt varnishes, 747
Assyrians, use of asphalt by, 25
Astatki, 431
Austria, asphalt in, 124, 203
distillation of, 280
extraction with water, 124
Australia:
asphalt in, 223
albertite in, 270
elaterite in, 263
impsonite in, 274
Australian caoutchouc, 263
Automobile:
acoustical compositions, 513
acoustical felts, 703, 714
brake-linings, 713
clutch-facings, 713
insulating-felt, 714
shim-strips, 714
tacking-strips, 714
Average diameter, definition of, 1061
Awnings, bituminized, 639
Azabache, 96

Babylonia, asphalt in, 5, 6, 30, 31, 32, 33, 41
Babylonians, use of asphalt by, 20
Bagasse tar, 280
Bagasse-tar pitch, 280
Bagga, 132
Baking japans, 753
Baking varnishes, 755
Baku, asphalt in, 39, 40
Ball-mill, 506
Barbados, glance pitch in, 237
Base-course of pavements, 583, 587
Base of bituminous-solvent compositions, 720
examination of, 1129
Base-sheet for roofs, 693
Batch stills, see "intermittent distillation"
Battery-box compound, 546
Battery boxes, 546, 553
Battery carbons, 368, 546
Baumé scale, 794
Bayberry wax, 543
Bedding-course of pavements, 597
Beechwood tar, 299
Beechwood-tar pitch, 300
Beeswax tar, 280
Beeswax-tar pitch, 280
Beet-residue tar, 280
Beet-residue-tar pitch, 280
Belting, bituminized, 546
Bentonite, 509
Benzol, solubility in, 965
Berginization, 75
Bergius process, 75
Bermudez asphalt, 50, 146
dehydration of, 148
mining of, 112, 148
molecular weight of, 990
properties of, 148
Biblical times, use of asphalt in, 5, 19, 42
Bibliography, 1183
Bibulous bodies, 546, 554
Binary bituminous mixtures, 481
Binder:
asphaltic, 568, 569, 570 (insert), 571
bituminous, 567
briquette, 491, 558
coal-tar, 570 (insert)
paving, 368, 431, 567, 570 (insert), 571
Binder course of pavements, 590
Bitosan pavement, 587
Bitoslag pavement, 587
Bitulithic pavement, 586
Bitume, 19
Bitumen Judaicum, 30
Bitumens:
associated minerals, 83
behavior under heat, 73
classification of, 44, 52, 62
composition of, 54
definition of, 42, 55
dehydration of, 114
depolymerization of, 72
dispersions of, 496, 503
distillation of, 119
early definition of, 42
effect of capillarity on, 88
effect of heat on, 72, 89
"elastic", 43

Bitumens—*Continued:*
erroneous use of term, 60
extraction of, 122
by means of solvents, 125
by means of water, 122
fluxing of, 478
geology of, 82
heating of, 115
impregnated rocks of, 86
lakes of, 85
metamorphosis of, 89
mining methods, 112
movement of, 87
occurrence of, 84
origin of, 44, 54, 82, 89
origin of word, 1
polymerization of, 72
pools of, 86
primary deposits of, 87
properties of, 54, 478, 778
pumping of, 119
refining of, 113
reservoirs of, 86
secondary deposits of, 88
sedimentation of, 122
seepages of, 84
shipping of, 113, 148, 185, 208
solubility of, 54, 525 (insert)
springs of, 84
storage of, 128
transportation of, 113
veins of, 84, 86
weather-resistance of, 478
word in:
Anglo Saxon, 1
German, 1
Latin, 1
Norwegian, 1
Sanskrit, 1
Bituminated cork mixtures, 552
Bituminated hair-felt, 705
Bituminated leather mixtures, 557
Bituminic substance, 97
Bituminized acoustical blocks, 719
Bituminized discrete aggregates, 1017
analysis of, 1044
examination of, 1017
sampling of, 773
tests for:
brittleness, 1024
coefficient of wear, 1025
compression, 1034
curling, 556
distortion under heat, 1037
effect of water on adhesion, 1018
electrical tests, 1044
extrusion, 1022
flammability, 556
flexural strength, 1036
flexure, 556
Bituminized discrete aggregates, tests for —*Continued:*
hardness, 1019
resistance to displacement, 1020
resistance to impact, 556, 1022, 1039
resistance to moisture, 1018, 1029
separation into component parts, 1044
shatter properties, 1024
softening-point, 1038
specific-gravity, 1017
tensile-strength, 1033
thickness, 1025
voids, 800, 1017
Bituminized blasting-fuses, 711
Bituminized brake-linings, 552, 713
Bituminized cement, 561
Bituminized clay articles, 546
Bituminized clutch-facings, 713
Bituminized conduits, 546, 713
Bituminized cords, 712
Bituminized fabrics, 617
analysis of, 1072, 1088
armored, 615
blistering of, 1155
checking of, 1154
cracking of, 1157
dusting of, 1157
grooving of, 1154
sampling of, 773
tests for:
bursting strength, 1078
electrical tests, 1086
entrapped air, 800, 1155
moisture, 1088
Mullen strength, 1078
pliability, 1080
resistance to dampness, 1084
resistance to heat, 1085
resistance to water, 1084
rubbing test, 1092
separation into component parts, 1088
special tests for insulating tape, 1086
tearing-strength, 1078
tensile-strength, 1077
thickness, 1077
voids, 800
weathering, 1153, 1167
weight, 1072
Bituminized felts for:
automobiles, 714
built-up roofs, 639, 642, 1072
expansion-joints, 639, 1072
floor-coverings, 639, 693, 1072
pipe-wrappings, 538
roofings and shingles, 639
sidings, 681
stucco-base, 719
waterproofing membranes, 639, 642, 697, 1072
Bituminized fiber-conduits, 713

Bituminized floor-coverings, 693
Bituminized insulating-board, 718
Bituminized mineral aggregates, 567, 1017
Bituminized papers, 703, 710, 1072
Bituminized pipes, 546
Bituminized plaster-board, 719
Bituminized ropes, 712
Bituminized shim-strips, 714
Bituminized sidings, 681
Bituminized stone, 546
Bituminized stucco-base, 714
Bituminized tacking-strips, 714
Bituminized tapes, 714, 1086
Bituminized tubes, 546
Bituminized wall-board, 717
Bituminized, wood-block pavements, 47 570 (insert), 598
Bituminized wood-pulp, 546
Bituminized wrapping-cloth, 640
Bituminous, definition of, 52, 61
Bituminous adhesives for:
  built-up roofs, 491, 493, 526, 628
  waterproofing, 491, 526, 697
Bituminous binder for pavements, 567, 570 (insert)
Bituminous broken-stone pavements, 583
Bituminous carpets, 578
Bituminous cement, 704, 744
  analysis of, 1117
  sampling of, 775
Bituminous coal, 97, 336
  classifications of, 55, 57, 62
  coking, 97, 336, 485
  composition of, 337, 345
  destructive distillation of, 340
  effect of heat on, 338, 343
  fluxing with tars, 485
  gas coals, 336
  high-temperature carbonization of, 337, 338
  low-temperature carbonization of, 338, 349
  metamorphosis of, 97
  non-coking, 347
  products derived from, 374 (insert)
  properties of, 337, 778
  volatile matter in, 282
Bituminous-coal shales, 276
  classification of, 55
  properties of, 778
Bituminous coating compositions, see "coating compositions"
Bituminous compositions, 526
Bituminous-concrete pavements, 570 (insert), 575, 586
Bituminous dispersions, 503, 1140
  apparatus used for making, 505
  bituminous substances used for, 508
  blowing of, 445
  breaking of, 507
Bituminous dispersions—*Continued:*
  coagulation of, 503
  continuous phase of, 503
  decrease the breaking speed of, 512
  discrete phase of, 503
  dispersing agents for, 508
  examination of, 1132
  external phase of, 503
  incorporation of asphalt solvents, 512, 742
  increasing the mobility of, 512
  increasing the speed of breaking, 512, 573
  internal phase of, 503
  labile, 511
  mixing types, 1138
  penetration types, 1138
  peptization of, 503
  preparation of, 505, 508
  quick-setting, 511, 1132, 1138
  semi-stable, 1132
  slow-setting, 511, 1132
  stable, 511, 1132
  types of, 503
  withstanding low temperatures, 512, 741
  tests for:
    analysis of, 1140
    behavior with aggregate, 1137
    calcium-chloride test, 1136
    demulsibility, 1136
    determination of dispersing agents, 1145
    determination of distillation residue, 1140
    determination of water and oils, 1143
    effects of freezing, 573
    examination of bituminous constituents, 1142
    ferrous-sulfate test, 1137
    homogeneity, 1133
    identification, 1132
    microscopic examination, 1133
    miscibility with water, 1139
    resistance to water, 1139
    sampling, 775
    settlement, 1135
    sieve test, 1133
    stability, 1135
    stone-coating, 1137
    viscosity, 1135
  uses for:
    anti-fouling paints, 741
    asphalt-fiber coatings, 741
    asphalt-plastic cement, 746
    automobile bodies, 513
    bituminous-concrete pavements, 589
    bituminous-macadam pavements, 584
    briquette binders, 513
    built up roofs, 688, 741
    cellulose-acetate lacquers, 753
    cement, 562

Bituminous dispersions, uses for—*Continued:*
coating paper, 710
coating roofings, 628
concrete, 562
cork compositions, 553
damp-proofing, 733
dust laying, 575
electrical purposes, 513
fibrated compositions, 554
filling cavities, 513
floors, 513
fungicides, 513
glazing concrete, 564
impregnating concrete, 546
impregnating felts, 512
impregnating papers, 512, 707
impregnating woven fabrics, 635
impregnating stone, 546
insecticides, 513
insulation purposes, 513
lubrication, 513
magnesium-oxy-chloride, 565
paints, 471, 513, 733, 741, 755
paving purposes, 512, 572, 575, 584, 589
pipe-coatings, 513
plastic-cements, 746
plaster-of-paris, 565
roofing purposes, 512
rubber substitutes, 513
shoe-fillers, 553
Sorel cement, 565
stucco-base coat, 741
tree-surgery, 513
wall-board, 717
waterproofing, 513, 562
Bituminous dust-laying compositions, 491, 570 (insert), 575
Bituminous emulsion paints, 471, 513, 733, 741, 755
Bituminous emulsions, see "bituminous dispersions"
Bituminous enamels, 750
for acid-proofing concrete, 534
for ships, 531
Bituminous expansion-joints, 493, 611, 1072
Bituminous fillers, 491, 574 (insert), 594, 595, 603
Bituminous fuels, 558
Bituminous-gravel pavements, 583
Bituminous grout, 531
examination of, 1017
Bituminous impregnating compositions, 491, 493
Bituminous insulating board, 718
Bituminous insulating compounds, 491, 493, 541, 552
Bituminous japans, 753
Bituminous joint-fillers, 594
Bituminous lacquers, 720
Bituminous lignite, 312
Bituminous macadam, 570 (insert), 575, 583
Bituminous matter:
discovered in New York State, 47
discovered in the United States, 47
Bituminous mixtures, 481, 489
carbonizing of, 493
classes of, 490
duplicating, 490
overheating of, 492
utilization of, 491, 493
Bituminous molding compositions, 493
Bituminous mortar, 531
Bituminous paints, see "bituminous-solvent compositions"
Bituminous paving materials, 491, 493, 567
Bituminous pipe-dips, 491, 493, 535
Bituminous pipe-sealing compounds, 491, 493, 535
Bituminous plaster-board, 719
Bituminous primer for ship-enamels, 532
Bituminous rail-fillers, 615
Bituminous revetment, 616
Bituminous rock, 56
Bituminous roofings, 491, 493, 1072
Bituminous rubber-substitutes, 491, 547
Bituminous saturation, see "impregnation"
Bituminous shingles, 658, 1072
Bituminous ship-enamel, 531
Bituminous sidings, 681
Bituminous-solvent compositions, 491, 493, 720, 1117
abrasion test for, 1120
acid-resisting, 737
adhesion test for, 1120
air-drying, 740
analysis of, 1117, 1130
analysis of dried films, 1131
anti-corrosive, 741
anti-fouling, 740, 741
asphalt, 720
asphalt-fibrous roof-coating, 737, 741
base of, 720
beer-cans, 740
bituminous, 720
checking of, 735
damp-proofing, 731, 732
early use of asphalt in, 16, 21, 31, 35, 41
electrical insulating, 740
emulsion, 471, 513, 741, 755
fillers for, 724
flooring compositions, 742
gelatinization of, 725, 727, 749, 753
grinding of, 731
manufacture of, 730
marine, 740, 741
masonry, 731
metal, 739

Bituminous-solvent compositions — *Continued:*
pigments for, 724
primer for roofing and waterproofing, 733
resistance to moisture, 1121
resistance to petroleum products, 729
roofing, 632, 734
sampling of, 775
solvent, 720
solvents for, 725
stone-backing, 733
stucco-base, 741
weathering of, 1158, 1167
wood, 739
tests for:
abrasion, 1120
acid-resistance, 739
adhesion, 1120
anti-corrosive properties, 723
color, 1120
dielectric strength, 740, 754, 1122
draining, 1118
drying, 1119
estimation of base, 1129
estimation of pigment and filler, 1128
estimation of solvent, 1123
examination of base, 1129
examination of pigment and filler, 1129
examination of solvent, 1128
flash-point, 1118
gloss, 1120
hardness, 1120
heat-resistance, 1121
hiding-power, 1120
mobility, 1118
plasticity, 1118
resistance to acids and alkalies, 1122
resistance to oil, 1122
separation into component parts, 1123
specific-gravity, 1117
spreading capacity and workability, 1118
viscosity, 1118
water-absorption, 1121
weather-resistance, 1158, 1167
Bituminous stucco-base, 719
Bituminous substances:
anti-oxidants, effect of, 722, 1128, 1154
associated mineral constituents, 70, 83
associated non-mineral constituents, 71
binary mixtures of, 481
blending of, 476, 483, 491
blistering of, 1155
calendering of, 551, 555
carbon in, 68
carbonization of, 338, 1149
changes induced by weathering, 1151
checking of, 1154
chemistry of, 65
Bituminous substances—*Continued:*
chlorinated, 80, 137, 301, 376, 444, 453, 542, 543, 628, 721, 752
classes of, 491
classification of, 42, 51
colloidal nature of, 66, 70
comminution of, 121, 158, 169, 201, 367, 565
complex mixtures of, 489
composition of, 54
composition of associated mineral constituents, 70, 83
composition of non-mineral constituents, 71
compounding of, 476, 483, 491
consistency of, 476
controlling fusibility and hardness of, 480
copper in, 272
correcting heterogeneity of, 484
cracking of, 72, 75
cut-back, 571
decreasing stickiness of, 489
decreasing susceptibility of, 486
definition of, 52
dehydration of, 114
depolymerization of, 72
discovery in New York State, 47
discovery in United States, 47
dispersions of, 503
distillation of, 119
distinguishing characteristics of, 778, 779
duplication of, 490
effect of heat on, 72, 89, 492
effect of moisture on, 1150
emulsification of, see "bituminous dispersions"
evaporation of, 1148
examination of, 776
extraction of, 122
by means of solvents, 125
by means of water, 122
extrusion of, 551, 554
fluxing of, 477, 478, 482, 491, 492
fusibility of, 477, 478
grooving of, 1154
hardening of, 80, 301, 376, 377, 402, 485
hardness of, 477, 478
heating of, 115, 491
hydrogenation of, 58, 74
hydrogen in, 68
improving amalgamation of, 483
incorporating fillers in, 1154
increasing adhesiveness of, 483
increasing fluidity of, 483
increasing softening-point of, 80, 485
increasing stability of, 485
increasing tensile-strength of, 487
increasing weather-resistance of, 487

Bituminous substances—*Continued:*
liquid, 52
lowering the softening-point of,, 482
mining methods, 112
mixtures of, 477, 481
mixtures with chlorinated rubber, 486, 541, 722
mixtures with rubber, 445, 453, 486, 487, 513, 541, 549, 613, 629, 722, 1055
molding of, 550
molecular composition of, 68
nitrogen in, 69
non-mineral matrix of, 65
origin of, 54
overheating of, 492
oxygenation of, 58, 74, 1148
oxygen in, 69
plastic range of, 1154
polymerization of, 72, 455, 714, 1150
precipitation products, 58, 125
preparing mixtures of, 477
properties of, 54, 478, 778
purity of, 493
reactions with:
acids, 58, 77, 78
alkalies, 58, 79, 1000
boron fluoride, 80
chlorine, 58, 80, 444, 453
formaldehyde, etc., 59, 79, 301, 375, 376, 377, 401, 486, 497, 721, 999
furfurol, 59
gases, 74, 444
halogens, 58, 80, 444, 453
hydrogen, 58, 74, 486
iodine, 58, 80
liquid sulfur dioxide, 77
metallic salts, 58, 81, 375, 497, 808
metalloids, 79
mineral acids, 58, 78, 401
nitric acid, 58, 78, 401, 486, 721
oxygen, 58, 74, 444
phosphoric acid, 58, 497, 509
phosphorus, 58, 80
phosphorus derivatives, 58
precipitation agents, 58
selenium, 80
sulfides, 453
sulfur, 58, 79, 453, 486, 549
sulfur derivatives, 58, 453
sulfur dichloride, 79, 375, 383, 453, 549
sulfur dioxide, 77
sulfuric acid, 58, 78, 401, 453, 486
sulfuric acid and formaldehyde, 79, 999
sulfur trioxide, 78
reducing susceptibility of, 486
references by Greeks and Romans, 27
refining of, 114
rendering wax-like, 489

Bituminous substances—*Continued:*
sampling of, 757, 762
sedimentation of, 122
semi-solid, 52
setting of, 1150
shipping methods, 113
softening of, 482
solid, 53
solubility of, 54, 514, 525, 525 (insert)
spontaneous hardening of, 1150, 1154
storage of, 128, 354
sulfur in, 68
sulfurized, 79, 301, 323, 375, 376, 401, 443, 452, 453
suspensions of, 503
terminology of, 51
tertiary mixtures of, 489
thixotropy of, 1150
transportation of, 113, 148, 185, 208, 354
ultimate analysis of, 68
utilization of, 493
volatility of, 477, 478
weather-resistance of, 477, 478
tests for:
acid-resistance, 534
acid value, 1001
adhesiveness, 855
aging index, 1150
anhydrides, 1002
anthraquinone reaction, 1015
appearance surface when aged, 784
asphaltenes, 1011
asphaltic constituents, 1007
asphaltic resins, 1011
asphaltous-acid anhydrides, 1011
breaking-point, 885
brittleness, 533
burning-point, 949
carbenes, 962
carbon, 982
chemical tests, 973
cholesterol, 1006
coefficient of expansion, 872, 875
cohesiveness, 854
colloidal capacity, 801
color in mass, 780
color in solution, 780
color value, 781
compression, 912
consistency, 837, 840
consistometer, 840
covering power, 532, 535
curling, 556
degasification, 877
degree of mercuration, 1000
diazo reaction, 477, 1013
diffusibility, 787
dimethyl sulfate test, 999
distillation test, 928
distinguishing characteristics, 778, 779

Bituminous substances, tests for—*Continued:*
ductility, 847
elasticity, 920
electrical resistance, 541
elemental composition, 982
entrapped air, 800, 877
ester value, 1002
evaporation, 924
fixed-carbon, 949
flammability, 556
flash-point, 935
flexure, 556
float test, 820
flow, 533
flow-point, 917
formolite reaction, 999
fracture, 984
free acids, 1001
free asphaltous acids, 1007
free carbon, 965
fusing-point, 895
glycerol, 1006
hardness, 828
heat content, 871
heterogeneity, 783
homogeneity, 525, 781
in solution, 782
to the eye, 782
under microscope, 782
when melted, 782
hydrogen, 982
impact, 556
indentation, 555
insoluble in:
benzol, 965
carbon disulfide, 951
carbon tetrachloride, 962
sulfuric acid, 995
water, 996
interfacial tension, 862
lactones, 1002
lactone value, 1002
Liebermann-Storch reaction, 1016
liquefying-point, 917
lustre, 785
mechanical tests, 809
melting-point, 913, 914
microscopic count of colloidal particles, 802
molecular weight, 990
naphthalene, 992
neutral fats, 1002
nitrogen, 988
oily constituents, 1012
oxygen, 989
paraffins, 992
penetration, 830
physical characteristics, 780
phytosteryl, 1006
plasticity, 828
properties of, 478
resilience of, 920
rubber mixtures, 1055
saponifiable constituents, 1000, 1004, 1006
saponification value, 1002
saturated hydrocarbons, 995
shatter test, 533
softening-point, 895
solidifying-point, 889
solid paraffins, 992
solubility tests, 525, 951
soluble in:
benzol, 965
carbon disulfide, 951
carbon tetrachloride, 962
petroleum naphtha, 964
sulfuric acid, 995
various other solvents, 970
specific-gravity, 788
specific-heat, 870
streak, 785
sulfonation residue, 995
sulfur, 984
surface-tension, 857
susceptibility index, 845
tar-acids, 991
tensile-strength, 854
thermal conductivity, 863
thermal expansion, 871
thermal tests, 863
thoroughness of blending, 480
twisting-point, 918
unsaponifiable constituents, 1006
unsaturated hydrocarbons, 995
viscosity, 809
voids, 800
volatile matter, 920
water, 973
water absorption, 785
weathering tests, 1147, 1151, 1159, 1167
workability, 533
xylene equivalent, 783
Bituminous surfacings, 578
plant-mix, 583
road-mix, 581
Bituminous suspensions, 501
Bituminous varnishes, 746
sampling of, 775
Bituminous wood-blocks, 598
Bitural, 376
Black amber, 96
Black-base of pavements, 587
Black grease, 394
Black oil, 431
Blast-furnace coal-tar, 279, 286, 347
associated mineral matter, 347
classification of, 57, 63

Blast-furnace coal-tar—*Continued:*
composition of, 338, 348
distillation of, 348, 354
distinguishing, 348
production of, 107, 347
properties of, 351, 778
recovery of, 347
yield of, 338
Blast-furnace coal-tar pitch, 371
classification of, 58, 63
production of, 107
properties of, 371, 372, 778
Blasting fuses, 711
Blau gas, 383
Blau-gas tar, 383, 384
Blending of bituminous substances, 476, 483
determining thoroughness of, 480
Blistering, 1155
Blisterwax, 132
Blocks, acoustical, 719
Blocks, asphalt, 570 (insert), 595
Blocks, asphalt-mastic, 605
Blower wax, 132
Blowing, effect on asphalt, 446, 1010
Blown Alberta asphalt, 179
Blown asphalt, see "blown petroleum asphalt"
Blown coal-tar pitch, 49, 374, 440
Blown fatty-acid pitch, 401, 440
Blown lignite tar and pitch, 323
Blown montan wax, 137
Blown native asphalt, 440
Blown Oklahoma asphalt, 166
Blown petroleum asphalt, 414, 440
acid value of, 451, 1009, 1010
advantages of, 446
asphaltic constituents in, 1009, 1010
carelessly prepared, 447
chlorinated, 444, 721
classification of, 56, 63
composition of, 452
continuous process, 444, 445
definition of, 431
discovery of, 49, 50, 440
distinguishing from residual asphalts, 450, 456
distinguishing from wurtzilite asphalt, 475
electrical resistance of, 543
first commercial production of, 441
fluxing of, 479
from asphaltic petroleum, 442, 443, 479
from naphthenic residual oils, 442
from non-asphaltic petroleum, 414, 442, 479
from pressure-tars, 428, 434, 437, 438, 465
froms residual oils, 440, 441, 442
from semi-asphaltic petroleum, 414, 442, 479
Blown petroleum asphalt—*Continued:*
from sludge asphalts, 440, 445, 464, 465
from sundry mixtures, 445
gases used, 444
intermittent process, 444
mechanical expedients, 445
oxidizing agents proposed, 443
properties of, 446, 449, 452, 479, 778
relation between:
softening-point and hardness, 446
softening-point and specific gravity, 449
removal of oily constituents, 448
saponification value of, 451
solubility of, 525 (insert)
sulfurized, 443
vacuum process, 442
water-absorption of, 786
weather-resistance of, 450, 479, 1171, 1174, 1178
Blown pressure-tar, 428, 434, 437, 438, 465
Blown residual oils, 433, 434
Blown sludge asphalt, 440, 446
Blown Texas asphalt, 170
Blown wood-tar pitch, 301
Blown wool-fat pitch, 440
Blue-gas, 379
Blue oil, 334
Blue rosin-oil, 303
Board, bituminized insulating, 718
Boat-decking, 653
Boats, early use of asphalt in, 14, 19, 20, 28, 31
Bog-head coal, 96, 306
Bog-head coal-tar, 336
Boiled tar, 292, 295
Bombiccite, 96
Bone-fat pitch, 386, 395, 402, 405
Bone grease, 396
Bone oil, 407
Bone tar, 279, 386, 407
classification of, 57, 63
composition of, 407
distillation of, 408
production of, 107, 407
properties of, 778
solubility of, 525 (insert)
yield of, 407
Bone-tar pitch, 386, 407
classification of, 58, 63
distillation of, 408
fluxing of, 408, 479
production of, 107, 407, 408
properties of, 408, 479, 778
solubility of, 408, 525 (insert)
weather-resistance of, 408, 479
Bottle-method of specific-gravity, 795
Brake-linings, 552, 713
Branchite, 96
Brattice cloth, 639

Brazil, asphalt in, 190
impsonite in, 273
Brea, 59
Breaking-point test, 885
Fraas method, 886
knife method, 886
Reeve and Yeager's method, 886
Breaking-weight of felts, 623
Brewers' pitch, 304
Bridge-planking, 616
Bridges, early use of asphalt in, 24
Briquette binders, 491, 558
Briquetting asphalt, 104
Brittleness test of paving mixtures, 1024
Brittleness test of ship-enamels, 533
Browncoal, 96, 311
Browncoal tar, see "lignite tar"
Brownian movement, 66, 81, 507, 524, 803
Broxburn retort, 329
Brunswick black, 739
B.S., 733
Bubble-cap tray-tower, 418, 420
Building blocks, 546
Building papers, 703
Built-up roofs, 47, 642, 685, 686, 693
adhesive compositions for, 526, 527, 693
primer for, 733
Bulk specific-gravity, 1068
Burgundy pitch, 304
Burial vaults, 557
Burlap, 625, 637, 639, 642, 653, 697
Burning oil, 76
Burning-point test, 949
Bursting-strength test, 1078, 1104
Buton (Boeton) Island, asphalt in, 224
Butyrellite, 96
Byerlite, 50, 441, 446

Cable-splicing compound, 546
Calcium-chloride test for dispersions, 1136
Calendering bituminous compositions, 551, 555
California, asphalt in, 141, 174
extraction with solvents, 125
extraction with water, 177
Calking, early use of asphalt for, 40, 42, 151, 191, 219
Calking compounds, 528, 565, 712
Canada:
albertite in, 267
asphalt in, 177
Candelilla wax, 543
Candle pitch, 386
Candle tar, 386
Cannel coal, 96, 275, 306
classification of, 57
Cannel-coal shale, 276
Cannel-coal tar, 336
Canvas, 625, 639
Caoutchouc, Australian, 263
Capillarity, effect on bitumens, 88
Cap-sheet for roofs, 687, 693
Carbenes, 66, 480, 962
Carbocoal tar, 350
Carboids, 266, 1008
Carbolic oil, 356, 370 (insert)
Carbol pitch, 281
Carbon, 68
colloidal, 66
determination of, 982
Carbon disulfide, solubility in, 951
colorimetric method, 955
Carbonization of bituminous substances, 338, 1149
Carbonization:
high-temperature, 338
low-temperature, 338
Carbon-oleosole, 66
Carbons for:
armatures, 368, 546
batteries, 368, 546
electric lights, 368, 546
electrodes, 547
Carbon tetrachloride, solubility in, 962
Carburetting oil, 380
Carburetted water-gas, 379
Carburetted water-gas tar, 286, 378, 778
Carburetted water-gas-tar pitch, 778
Carnauba wax, 543
Carpet-coat for pavements, 578
Carpeting medium, 431
Cascade system of dehydration, 353, 362
Castings, see "core compounds"
Cataphoresis, 81
Cattle sprays, 366
Cellophane, use of, 539, 710, 713
Cellular asphalt, 613
Cellular pitch-coke, 370
Celluloid, use of, 722
Cellulose acetate, see "cellulose-ester lacquers"
Cellulose-ester lacquers, 751
Cellulose nitrate, see "cellulose-ester lacquers"
Cement:
asphaltic, 570
asphalt-plastic, 744
bituminized, 561
bituminous, 703, 744
lap, for roofings, 683, 734
plastic-slate, 528
roofing, 693
Cement-waterproofing compounds, 491
Central America, glance pitch in, 241
Centrifugal method of:
dehydrating coal tars, 352
dehydrating lignite tars, 317
dehydrating petroleum, 411
separating bituminous matter, 1050
Cerecloth, 707

Ceresine, 56, 62, 131
adulteration of, 132
classification of, 56
electrical resistance of, 542, 543
molecular weight of, 995
refractive index of, 995
solubility of, 525 (insert)
Chapapote, 59, 144
Charms, early use of asphalt in, 39
Charpy impact-test, 1040
Chaser, 506
Checking:
of paints, 735
of roofings, 1154
Cheese pitch, 189
Chemical tests, 973
Chemistry of:
asphalts, 46
bituminous substances, 65
Chemoasphalte, 58
Chemopeche, 58
Chewing-gum, use of asphalt in, 49
Chilean mill, 506
China, asphalt in, 221
Chinese wax, 543
Chlorinated bituminous substances, 58, 80, 137, 301, 376, 444, 453, 542, 543, 628, 721, 752
Chlorine, reactions with bituminous substances, 58, 80
Cholesterol, 1006
Cholesterol pitch, 386, 399
Chrysotile asbestos, 624
Classification of:
asphalts, 56, 62
bitumens, 44, 55, 62
bituminous substances, 51, 62, 63
pyrobitumens, 62
pyrogenous distillates, 63
pyrogenous residues, 63
Clay articles, bituminized, 546
Clay dispersion test, 802
Clay pigeons, 368, 547
Cleats, roofing, 683
Cleveland test for flash-point, 941
Clutch-facings, 552, 713
Coagulation, 503
Coal:
Albert, 48, 267
anthracite, 97
asphaltic, 47
bituminous, 97, 336
bog-head, 96, 306, 336
cannel, 96, 306, 336
coking, 97, 336, 345, 485
distillation, 312
distillation of, 279, 340, 345, 347, 348, 379
fire, 312
gas, 336
geology of, 82
Coal—*Continued:*
non-coking, 345
pitch, 143
products derived from, 370 (insert)
Coal gas, 45, 339, 342
Coalite process, 338, 348
Coalite tar, 349, 350
Coal shales, 62, 94, 276, 277, 778
Coal tar, 336
blast-furnace, see "blast-furnace coal tar"
classification of, 57, 63
coefficient of expansion of, 875
coke-oven, see "coke-oven coal tar"
composition of, 337
cracking of, 354, 358, 376, 429
dehydration of, 344, 350
deodorizing of, 375
discovery of, 43
dispersions of, 367, 496, 503
distillation of, 355, 357, 359, 360, 363, 364
distinguishing from oil-gas tar, 385
formaldehyde condensation product, 376
gas-works, see "gas-works coal tar"
hardening of, 376
hydrogenation of, 777
low-temperature, 338, 348, 351, 778
mixtures with:
asphalt, 477, 484
asphaltic pyrobituminous shales, 276, 485
chlorinated rubber, 486
coke-oven tar, 439
native asphalts, 484
oil shales, 485
petroleum asphalts, 484
pressure tar, 439
rubber, 486
Trinidad asphalt, 484
nitrated, 721, 739
oxidized, 49, 374, 440
oxidizing of, 49
producer-gas, see "producer-gas coal-tar"
production of, 45
products from, 356, 370 (insert)
properties of, 338, 350, 351, 778
refined, 367, 370 (insert)
refining of, 44, 355, 365
solubility of, 525 (insert)
solvents from, 45, 125, 365, 370 (insert), 514, 515, 522, 725
stills for, 355
storage of, 354
surface-tension of, 521
transportation of, 354
yield of, 338
Coal-tar binders, 567, 570 (insert)
Coal-tar distillate oil, 601

Coal-tar distillates, 125, 365, 520
Coal-tar paving binders, 570 (insert)
Coal-tar paving oil, 601
Coal-tar pitch, 336
  blast-furnace, see "blast-furnace coal-tar pitch"
  blown, 49, 374, 440
  characteristics of, 373
  chlorinated, 80, 376
  classification of, 58, 63
  coefficient of expansion of, 875
  coke-oven, see "coke-oven coal-tar pitch"
  comminuted, 367, 499
  cooler for, 360, 367
  crystallization inhibited, 368
  cut-back, 367, 482, 571
  deodorizing of, 375
  discovery of, 43
  dispersions of, 367, 496, 503
  dissolving oil-shales in, 485
  distillation of, 368, 369, 370
  distinguishing between high and low-temperature pitches, 374
  distinguishing from:
    asphalt, 524, 997
    oil-gas-tar pitch, 384
    water-gas-tar pitch, 384
  fluxing of, 367, 479, 484
  free-carbon removal, 367, 542, 729, 740
  free-carbon settlement, 721, 729
  gas-works, see "gas-works coal-tar pitch"
  handling of, 367
  hard, 370 (insert)
  low-temperature, 371, 778
  medium, 370 (insert)
  mixtures with:
    asphalt, 477, 484
    asphaltic pyrobituminous shales, 485
    chlorinated rubber, 486
    native asphalts, 484
    oil shales, 485
    pressure-tar, 484
    rubber, 486
    Trinidad asphalt, 484
  molding of, 367
  producer-gas, see "producer-gas coal-tar pitch"
  production of, 109
  properties of, 370, 479, 778
  recovery of, 367
  rendering insoluble, 729
  resins in, 369
  soft, 370 (insert)
  solubility of, 525 (insert), 729
  straight-run, 367
  sulfonated, 375
  sulfurized, 375, 527, 549
  surface-tension of, 521
  suspensions of, 367
Coal-tar pitch—*Continued:*
  water-resistance of, 368, 698
  weather resistance of, 368, 779
Coal-tar residuals, 336
Coal-tar saturated-felt, 639, 642, 693
Coal-tar solvents, 45, 125, 365, 370 (insert), 522, 725
Coating compositions for:
  blasting-fuses, 711
  concrete, 534, 737
  conduits, 713
  electrical tape, 715
  electrical wires, 712
  flooring, 696, 742
  insulating board, 718
  masonry, 731, 733
  metal, 739, 741
  papers, 707
  pigmented, 631
  pipes, 368, 535
  plaster-board, 719
  roofings and shingles, 628, 734, 737
  ships, 531, 740
  stucco-base, 719
  wood, 739, 741
Coating test for bituminous dispersions, 1137
Coefficient of expansion, 406, 872, 875
Coefficient of wear test, 1025
Cohesion test, 854
Cohesiveness, 854
Coke, 282, 283, 284, 320, 322, 344, 370 (insert)
  acid, 460, 463
  petroleum, 416, 417, 428
  pitch, 369
  yield of, 282
Coke-oven coal-tar, 279, 344
  classification of, 57, 63
  coefficient of expansion of, 875
  collector-main recovery of, 364
  composition of, 346
  cracking of, 429
  distillation of, 354
  hydrogenation of, 77
  mixture with pressure tar, 439, 484
  production of, 106, 344
  properties of, 338, 351, 778
  recovery of, 346
  resinous bodies from, 346, 369
  retorts used, 345
  yield of, 338, 345, 346
Coke-oven coal-tar pitch, 371
  classification of, 58, 63
  collector-main recovery of, 365
  properties of, 371, 372, 778
Coke-oven lignite-tar, 317
Coke ovens:
  beehive, 344
  by-products' recovery, 344

Cokey pitch, 190
Coking coal, 97, 336, 345, 485
Cold tars, 578
Collecting oils, 559
Collector-main condenser, 364
Collodion, see "cellulose-ester lacquers"
Colloidal capacity test, 801
  clay dispersion method, 802
  microscopic count method, 802
Colloidal carbon in asphalts, 66
Colloidal fuel, 558
Colloidal mineral particles, 70
Colloidal nature of asphalt, 66, 70, 93
Colloidal particles, 66, 496, 808
  admixed mechanically, 496
  inorganic, 496, 497
  liberated "in situ," 497, 501
  organic, 496, 497
Colloid, protective, 503
Colloid mill, 505, 506
  beater type, 507
  Hurrell, 507
  Plauson, 507
  smooth-surfaced type, 507
Colloids, 504
  hydrophile, 504
  hydrophobe, 504
  lyophile, 504
  lyophobe, 504
  protective, 504
  reversible, 504
Colombia:
  asphalt in, 191
  asphaltite in, 241
  glance pitch in, 241
Color:
  in mass, 780
  in solution, 780
  of roofings, 1157
  value, 781
Colorado:
  grahamite in, 252
Colored pigments, see "pigments"
Color test for bituminous-solvent compositions, 1120
Colprovia pavement, 587
Combustion, partial, 286
Comminuted asphalt, 121, 158, 169, 201, 565
Comminuted pitch, 367
Compacting weight of fillers, 1172
Complex bituminous mixtures, 489
Compositions or compounds:
  acid-proofing concrete, 534
  adhesive, 526, 628, 697
  artificial lumber, 557
  asphalt-cork, 552
  asphalt-leather, 557
  battery-box, 546, 553
  bituminated-leather, 557
Compositions or compounds—*Continued:*
  brake-linings, 552
  briquette binders, 558
  built-up roofing, 526, 527, 693
  burial vaults, 557
  cable-splicing, 546
  calking, 528, 565, 712
  cement-waterproofing, 534, 560
  clutch-facings, 552
  coating, see "coating compositions"
  conduits, 557
  core, 368, 558
  damp-proofing, 526
  dust-guards, 550
  dust-laying, 491, 570 (insert), 575
  electrical, 368, 491, 493, 541, 542, 547, 550
  fibrated, 554
  filling teeth, 550
  flooring, 742
  floor tiles, 555
  floor treads, 557
  friction elements, 552
  golf courses, 554
  grout, 531
  handles, 550
  insoles, 553
  insulating, 491, 493, 541, 552
  joints, 551, 554
  junction-box, 546
  knobs, 550
  molded, 550
  paving, 368, 574
  phonograph records, 557
  pigmented, 631
  pipe-dips, 368, 491, 493, 535
  pipes, 557
  pipe-sealing, 491, 493, 535, 539
  plastic, 528, 744
  pot-head, 546
  push-buttons, 550
  railroad ties, 557
  roof-copings, 555
  roofing, 491, 493
  roof tiles, 554
  rubber substitutes, 547
  saturating, see "saturating compositions"
  ship-enamel, 531
  shoe-fillers, 553
  sky-lights, 551
  small molded articles, 550
  step-treads, 557
  tree-surgery, 554
  vacuum impregnating, 542
  washers, 551
  waterproofing, 368, 491, 526, 560, 697
Composition roofings, 44, 47
Compounding of bituminous substances, 476
Compounds, see "compositions"

Compressed asphalt pavements, 589
Compression test of softening-point, 912
Compressive strength of molded compositions, 1034
Concrete, acid-proofing of, 737
Concrete, bituminous, 570 (insert), 575, 586
Concrete waterproofing, 534, 560
Condensed asphalt, 431
Condenser, collector-main, 364
Conductivity test for weathering, 1169
Conduits, bituminized-fiber, 546, 713
  molded, 557
Consistency, definition of, 828
Consistency of bituminous substances, 476
Consistency tester, 837, 840
Consistency tests, 828
  consistometer, 840
  Moh's scale, 829
  needle penetrometer, 830
Consistometer, 840
Continuous distillation of:
  coal, 340, 347, 348
  coal tar, 362
  fatty acids, 388
  hardwood, 293
  lignite, 313, 316
  lignite tar, 319
  petroleum, 417
  shale, 324
  shale tar, 332
  wood tar, 292, 293
Continuous phase, 503, 504
Cooler for:
  asphalt, 413
  pitch, 360, 367
Coorongite, 263
Coorongitic shale, 331
Cords, bituminized, 712
Core compounds, 558
Cork, bituminated, 552
Cork tar, 280
Cork-tar pitch, 280
Corn-oil-foots pitch, 386, 395, 402, 405
Corn-oil pitch, 386, 395
Cotton-oil pitch, 386, 395
Cotton pitch, 386, 395
  solubility of, 525 (insert)
Cotton-seed foots, 386
Cotton-seed-foots pitch, 386, 402
Cotton-seed-oil-foots pitch, 386, 395, 405
Cotton-seed-oil pitch, 386, 395
Cotton-stearin pitch, 386, 395
Coumarone resin, 483, 487, 509, 530, 543, 721, 729, 736, 749, 753
Course of pavements:
  base, 583, 587
  bedding, 597
  binder, 590
  foundation, 583, 587, 590, 597, 602
Course of pavements—*Continued:*
  surface, 578, 584, 588, 590
  wearing, 581, 583
Covering, bituminized floor, 693
Covering capacity of:
  acid-proof enamel and varnish, 535
  bituminous-solvent compositions, 1120
  ship-enamel, 532
Covering, teredo-proof, 620
Covering, wire, 712
Cracked distillate, 414, 416
Cracked residuum, 438
Cracking-coil asphalts, 438, 439
Cracking distillation, 414
Cracking of:
  asphalt, 424, 428, 438, 453
  asphaltic pyrobitumens, 72, 472
  bituminous substances, 72, 75
  coal tar, 354, 358
  petroleum products, 278, 279, 286, 416, 417, 424
  tars, 238, 319, 358, 376, 429
  wurtzilite, 472
Cracking processes, 424, 428
  Burton, 426
  Cross, 425
  Dubbs, 428
  Holmes-Manly, 426
  liquid-phase, 424
  mixed-phase, 426
  tube-and-tank, 424
Cracking-still tar, 435
Crackwax, 132
Creosote or creosote oil from:
  blast-furnace tar, 348
  coal tar, 356, 364, 366, 370 (insert)
  coke-oven tar, 365
  lignite tar, 315, 316, 320, 321
  low-temperature tar, 349
  peat tar, 308, 309
  wood tar, 290, 293, 295
Creosote-coal-tar solution, 600
Creosote primer, 698, 733
Creosote preservatives, 47, 600, 601
Cresol, 366
Cresol pitch, 58, 281, 377
  solubility of, 377
  uses of, 377
Crude naphtha, 365, 366
Crude scale-wax, 415, 416
Crude wax, 415, 416
Cuba:
  asphalt in, 144, 180
  discovery of asphalt in, 41
  glance pitch in, 238
  grahamite in, 254
Cube method of softening-point, 908
Culm, 499, 558
Cumarone pitch, 281
Cumar resin, see "coumarone resin"

Curling, test for, 556
Cushion layer of pavements, 597, 602
Cut-back asphalt, 104, 414, 431, 571
Cut-back coal-tar pitch, 367, 482, 571
Cut-back products, coefficient of expansion of, 875
Cut-back residual oil, 414, 433
Cwidu, 1
Cycle stock, 424
Cyclic hydrocarbons, 349, 409
Cylinder oil, 415
Cylinder stock, 414, 422, 424, 430
Czecho-Slovakia, asphalt in, 204

Damp-course, 609
Dampness, resistance of bituminized fabrics to, 1084
Damp-proofing coatings, 731, 732
Damp-proofing compositions, 526
Damp-proofing methods, 702, 704, 705
Dead oil, 366
Dead sea:
  asphalt in, 19, 29, 30, 32, 33, 35, 36, 40, 88
  glance pitch in, 243
Deck cloth, 653
Decking for boats, 653
Decorative roll-roofings, 655
Definition of:
  A. C., 570
  adhesion (adhesiveness), 855
  aggregate, 1060
  amicrons, 803
  asphalt, 56
  asphalt cement, 570
  asphaltenes, 964
  asphaltic pyrobitumen, 57
  asphaltite, 56
  asphalt mastic, 604
  asphalt-mastic cake, 605
  average diameter, 1061
  bitumen, 55
  bituminous, 52, 61
  bituminous grout, 531
  bituminous substance, 51, 52
    liquid, 52
    semi-solid, 52
    solid, 53
  blown petroleum asphalt, 431
  briquetting asphalt, 104
  carbenes, 962
  cohesion (cohesiveness), 854
  consistency, 828
  continuous phase, 503
  creosote-coal-tar solution, 600
  creosote preservative, 600
  cut-back asphalt, 104
  cut-back coal-tar pitch, 367
  damp-proofing, 704
  deflocculating agent, 503
Definition of—*Continued:*
  density, 790
  discrete phase, 503, 504
  dispersing agent, 503
  dispersion, 1060
  emulsified asphalt, 104
  emulsifying agent, 503
  engine distillate, 430
  external phase, 503
  fatty-acid pitch, 386
  fixed-carbon, 949
  flocculate, 1060
  flux or fluxing oil, 104, 482
  free carbon, 965
  fluel oil, 430
  fusing-point, 895
  gas oil, 430
  gasoline, 429
  gel, 503
  hydrophile, 504
  hydrophobe, 504
  hydrosol, 503
  individual particles, 1060
  internal phase, 503
  joint-filler, 594
  kerosene, 430
  lubricating oil, 430
  lyophile, 504
  lyophobe, 504
  malthenes, 964
  mastic, 104
  mastic cake, 104
  melting-point, 895
  microns, 803
  mineral aggregate, 574
  mineral rubber, 547
  mineral wax, 55
  mobility, 828
  molding compounds, 104
  native asphalt, 56
  non-asphaltic pyrobitumen, 57
  non-uniformity, 1061
  oil shale, 275
  oleosol, 503
  paraffin wax, 431
  paving asphalt, 104
  penetration, 833
  peptizing agent, 503
  petrolatum, 430
  petrolenes, 964
  petroleum, 55
    asphaltic, 55, 409
    non-asphaltic, 55, 409
    semi-asphaltic, 55, 409
  petroleum naphtha, 429
  petroleum spirits, 429, 430
  phase, 503
  pipe-coating, 104
  pitch, 58
  plasticity, 828

Definition of—*Continued:*
poise, 817
protective colloid, 503
pyrobitumen, 55
pyrogenous, 52
pyrogenous asphalt, 56
refined asphalt, 570
refined coal-tar, 367
residual asphalt, 431
residual oil, 431
road oil, 104
rock asphalt, 62, 70
roofing asphalt, 104
roofing flux, 431
samples, types of, 758, 760
sludge asphalt, 431
softening-point, 895
sol, 503
specific-gravity, 788, 789
absolute, 788
apparent, 789
bulk, 790
stabilizing agent, 503
straight-run asphalt, 414
straight-run coal-tar pitch, 367
sub-microns, 803
sulfurized asphalt, 431
suspension, 503
tar, 57
thermal conductivity, 866
tops, 430
ultimate working unit, 1060
ultra-microns, 803
waterproofing, 704
integral, 696
membrane, 696
waterproofing asphalt, 104
wax tailings, 431
yield value, 828
Deflocculating agent, 503
Degasification test, 877
Dégras, 398
Dégras oil, 399
Dégras stearin, 399
Degree of mercuration, 1000
Dehydration of:
asphalt, 114
coal tar, 344, 350
lignite tar, 315, 317
native asphalts, 114
peat, 307
peat tar, 308
petroleum, 411, 418
shale tar, 332
Trinidad asphalt, 114, 187
water-gas tar, 380
wood tar, 293
Dehydration processes:
cascade system, 353
centrifuging, 352, 411
Dehydration processes—*Continued:*
distilling, 353
settling, 350, 352, 411
tube-heaters, 353, 412, 418
Delmonte tar, 350
Demulsibility test for bituminous dispersions, 1136
Density, see "specific-gravity"
Densometer, 1107
Depolymerization of:
albertite, 72, 473
asphalt, 72
asphaltic pyrobitumens, 72
bituminous substances, 72
elaterite, 72
grahamite, 259
impsonite, 72
pyrobituminous shales, 276
wurtzilite, 72, 472
Deposits of bitumens:
primary, 87
secondary, 88
Derna asphalt, 204
Destructive distillation:
condensing system, 284
cracking, 283
oxygen content, 282
pressure, 284
temperature, 282
time of heating, 283
volatile constituents, 281
Destructive distillation of:
asphaltic petroleum, 412, 414
asphalts, 39, 40, 118, 119, 203, 204, 206, 209, 280
bones, 279, 407
bone-tar pitch, 408
coal, 279, 340, 345, 347, 348
fatty acids, 398
general, 281
hardwood, 279, 290
lignite, 279, 313, 316
non-asphaltic petroleum, 416
ozokerite, 130
peat, 279, 307
petroleum, 279, 412, 414, 416
rosin, 302, 303
semi-asphaltic petroleum, 416
shale, 277, 279, 324
soft wood, 279, 296
wurtzilite, 472
Diazo reaction, 477, 1013
Diethyl sulfate test, 999
Diffusibility test, 787
Dimethyl sulfate test, 999
Dinite, 96
Diphenyl:
use for fluxing, 483
use in heating, 117
Dippel oil, 407, 1147

Discrete aggregates, 494
Discrete phase, 503, 504
Disinfectants, 366
Disintegration, 505
Dispersing agents, 503, 509
  classes of 496
  combinations of, 511
  determination of, 1145
  drying of, 496
  filiform, 504
  hydrophile, 504
  hydrophobe, 504
  incorporation of, 494
  inorganic, 509
  lyophile, 504
  lyophobe, 504
  organic, 509
  reversible, 504
  water-soluble, 511
Dispersion, 1060
Dispersion mill, 506
Dispersions, see "bituminous dispersions"
Dispersions of bituminous substances with:
  colloidal solids, 496, 497, 509, 510
  water, 503
Dispersions of clay, 509, 802
Dispersion test, 802
Dispersoids, 81
Displacement test for paving compositions, 1020
Distillate oil, 600
Distillates:
  coal-tar, 125, 365, 520
  lignite-tar, 319
  peat-tar, 309
  petroleum, 429
  pyrogenous, 52, 63, 478, 525 (insert)
  rosin, 303
  shale-tar, 333
  wood-tar, 292, 294, 296
Distillation:
  continuous, see "continuous distillation"
  cracking, 414
  destructive, see "destructive distillation"
  dry, see "dry distillation"
  early use of asphalt for, 39, 40
  fractional, see "fractional distillation"
  gas-recirculation, 361
  Henderson process of, 332
  intermittent, see "intermittent distillation"
  steam, see "steam distillation"
  steam with vacuum, 359
  straight, 414
  temperature of, 282
  vacuum, see "vacuum distillation"
Distillation of:
  animal oils, 388
  asphalts, 39, 40, 118, 119, 203, 204, 206, 209, 380
Distillation of—*Continued:*
  bones, 297, 407
  bone tar, 408
  coal, 279, 340, 345, 347, 348, 379
  coal tar, 355, 357, 359, 360, 362, 363, 364
  coal-tar pitch, 369
  fatty acids, 388, 389, 390, 395, 398
  gilsonite, 230
  hardwood, 279, 290, 293
  lignite, 279, 313, 316
  lignite tar, 319, 320
  montan wax, 137
  ozokerite, 130
  peat, 279, 307
  peat tar, 309, 310
  petroleum, 279, 412, 414, 415, 416, 417, 418
  pyrobituminous shales, 277
  rosin, 301, 302, 303
  shale, 277, 279, 324
  shale tar, 332
  soft wood, 279, 295, 296
  vegetable oils, 388
  wood, 279, 290, 293, 295, 296
  wood tar, 292, 293, 296
  wool grease, 399
Distillation coal, 312
Distillation test, 928
  for bituminous dispersions, 1140
  for coal-tar products, 934
  for cut-back asphaltic products, 931
  for road oils, 928
Distilled-grease olein, 399
Distortion under heat test for molded products, 1036
Dopplerite, 96
Dow ductility test, 848
Draining test for bituminous-solvent compositions, 1118
Dried paint films, analysis of, 1131
Drill, 625, 639
Dry distillation of:
  asphaltic petroleum, 412, 414
  asphalts, 39, 40, 118, 119, 203, 204, 206, 209, 280
  bones, 407
  coal, 340, 345, 347
  gilsonite, 230
  hardwood, 290
  lignite, 313, 316
  lignite tar, 319
  non-asphaltic petroleum, 416
  ozokerite, 130
  peat, 307
  peat tar, 309
  petroleum, 412, 414, 416
  rosin, 301
  semi-asphaltic petroleum, 416
  shales, 277

Dry distillation of—*Continued:*
soft wood, 296
wurtzilite, 472
Drying test for bituminous-solvent compositions, 1119
Dubb's asphalt, 431, 452
Duck, 625, 639, 654
Ductility test, 847
author's method, 852
Dow's method, 848
Durability of:
paints, 1159
roofings 652, 1158
Dust, asphalt, 565
Dust-catchers, 347
Dusting, 1157
Dust-laying compositions, 491, 570 (insert), 575
Dust-laying oil, 431, 570 (insert), 575
Dust palliatives, 575
Dust preventatives, 575
Dutch East Indies, asphalt in, 224
Duxite, 96
Dysodile, 96

Earth wax, see "mineral wax"
Ebano, 446
Ebonite, 549
Ebonized lumber, 546
Ecuador, asphalt in, 191
Egypt, asphalt in, 221
Egyptians, use of asphalt by, 16
Elastic bitumen, 43
Elasticity, test for, 920
Elaterite, 263
classification of, 55, 57, 62
depolymerization of, 72
discovery of, 43, 263
metamorphosis of, 95, 262
properties of, 262, 778
Elaterite in:
Asia, 263
Asiatic Russia, 263
Australia, 263
England, 263
Europe, 263
Electrical insulating compounds, 368, 491, 493, 541, 547, 550
Electrical insulating japans, 753, 754
Electrical insulating paints, 740
Electrical insulating papers, 709
Electrical insulating tape, 714, 1086
Electrically insulated wires, 712
Electrical resistance of:
asphalt, 541, 542, 543
fabrics, 1086
paints, 740, 754, 755, 1122
papers, 709
Electrical tests for:
bituminized fabrics, 1086
Electrical tests for—*Continued:*
bituminous-solvent compositions, 1122
molded compositions, 1044
Electrical varnishes, 746, 753
Electric-light carbons, 368, 546
Electrodes, 547
Elemental composition of bituminous substances, 982
Elutriation test, 1058
Embalming, use of asphalt for, 17, 33
Embankments, use of asphalt in, 26, 27, 616
Emulsified asphalt, 104
Emulsifying agent, 503
Emulsion japans, 755
Emulsion of water-gas tar, 380
Emulsion paints, 513, 741
Emulsions, see "dispersions"
Enamels:
acid-proof, 534
bituminous, 750
ship, 531
Enamels for:
acid-proofing concrete, 534
ships, 531
Engine distillate, 430
England, elaterite in, 263
Engler viscosity, 811
Enriching oil, 380
Entrapped air, test for, 800, 877
Equi-viscous temperature, test for, 810
Ester value, 1002
Esto-asphalt, 335
Esto-bitumen, 335
Ethyl-ether, solubility in, 971, 973
Euosmite, 96
Europe:
albertite in, 269
asphalt in, 150, 191
elaterite in, 263
glance pitch in, 242
ozokerite in, 132
Evaporation of:
bituminous substances, 1148
solvents, 726
Evaporation test, 924
Examination of:
bituminized fabrics, 1072
bituminized discrete aggregates, 1017
bituminous dispersions, 1132
bituminous-solvent compositions, 1117
bituminous substances, 776
Exothermic reaction, 76
Expansion, coefficient of, 406, 872, 875
Expansion-joints, 491, 493, 611
Explosives, 565
Exposure tests, 1153
Extenders, 548
External phase, 503, 504

Extraction methods:
centrifugal, 1050
cold, 1048
of aggregates with asphaltic binder, 1045
of aggregates with coal-tar pitch binder, 1053
hot, 1045
Extraction with solvents of:
asphalt, 125
lignite, 128, 136, 313
montan wax, 128, 136, 313, 314
pyropissite, 128, 136
retort lignite, 314
roofing waste, 128
wood, 297
Extraction with water of:
asphalts, 122, 165, 204
ozokerite, 124, 130, 134
roofing waste, 554
Extractors, 124, 967, 970, 1047, 1050, 1054
Extrusion of bituminous substances, 551, 554
Extrusion test for paving compositions, 1022

Fabrics:
analysis of, 1072, 1088, 1111
armored, 615
asbestos, 624, 642, 646, 660
bituminized, 617
building, 702
felted, 617
flooring, 693
insulating, 709, 714
mulching, 710
roofing, 617
sampling of, 773
saturated, 641
saturating of, 634
testing of, 1072
waterproofing, 642, 697, 700, 703
woven, 625, 698
wrapping, 709
Factice, 488
Factis, 548
Faktis, 548
Falkland Islands, albertite in, 269
Falling-ball viscosity test, 823
Falling coaxial-cylinder viscosity test, 826, 845, 1150
Fastening devices for roofing 682
Fat pitch, 386
Fats, neutral, 1002
Fatty-acid distillation, 388, 389, 390, 395, 396, 398
Fatty-acid pitch, 386
behavior on heating, 401
blown, 401, 440
Fatty-acid pitch—*Continued:*
classification of, 58, 63
coefficient of expansion of, 406
composition of, 400
distinguishing from:
asphalts, 403
wurtzilite pitch, 475
effect of heat on, 401
electrical resistance of, 543
flame-resisting properties of, 713
fluxing of, 406, 479
fluxing with wood-tar pitch, 301
formaldehyde condensation product, 401
from bone-fat, 396, 405
from corn-oil foots, 395, 405
from cotton-seed-oil foots, 394, 405
from fats and oils, 387, 393
from garbage, 397, 405
from lard, 396, 402, 404
from packing-house refuse, 395, 404
from palm-oil, 402, 405
from sewage, 397, 405
from tallow, 395, 402, 404
from woolen-mill waste, 397
from wool-grease, 398, 405
hardening of, 401, 402
identification of, 1130
molecular weight of, 400
nitrated, 401
production of, 107, 386, 387
properties of, 399, 402, 404, 405, 479, 778
reactions with acids, 401
rubber pitch, 400, 401
saponifiable constituents in, 400, 402
saponification value of, 403
solubility of, 401, 525 (insert)
sulfurized, 401, 403, 721
weather-resistance of, 400, 406, 479
Felt:
acoustical, 703, 714
air-resistance of, 1107
analysis of, 1111
asbestos, 624, 642, 646, 660
asphalt-saturated, 634, 642, 693, 697
breaking-weight of, 623
coal-tar saturated, 639, 643, 693
examination of, 1099
fiber composition, test for, 1111
fireproofing of, 623
flax, 703
flooring, 693
hair, 703
insulating, 714
kerosene test for, 1110
microscopical examination of, 1111
moisture content of, 1103
"number" of, 622, 1102
oil-penetration test for, 1109
porosity of, 1107
rag, 618, 642

Felt—*Continued:*
roofing, 617, 642
saturating, 634
saturating-capacity, test for, 1110
saturating-speed test for, 1109
slaters', 639
sound-proofing, 714
strength test for, 1105
string, 624
tarred, 639, 642, 693
thickness test for, 1103
threaded, 624
waterproofing, 642
weight test for, 1102
Felted fabric, 1072
Fermentation-residue tar, 280
Fermentation-residue-tar pitch, 280, 281
Ferrous-sulfate test for bituminous dispersions, 1137
Fettpech, 386
Feuerkohle, 312
Fiber composition of felt and paper, 1111
Fiber conduits, 713
Fibers, 499
asbestos, 624
combinations of, 622
inorganic, 499, 622
organic, 499, 621, 1071
Fibers present in:
felt, 619, 1111
paper, 705, 1111
test for ascertaining, 1111
Fiber-wax, 132
Fibrated bituminous compositions, 554
Fibrous fillers, 499, 613
Fibrous wax, 132
Fichtelite, 96
Filaments of asphalt, 565
Filbertine pavement, 587
Fillers, 630
adsorptive capacity of, 1066
air-separation test for, 1060
asphaltic, 491, 570 (insert), 594, 603
bituminous, 491, 574 (insert), 595, 603
effect on weathering, 1154
examination of, 1055, 1129
fibrous, 499, 613
finely-divided, 494
granulated, 613
incorporation of, 494
inorganic, 497, 613
joint, 570 (insert), 574
mineral, 486, 488, 496, 497, 574 (insert), 724
organic, 499, 613, 1071
physical properties of, 1176
pigment, 631, 724
pitch, 594, 603
powdered, 497
rail, 615
Fillers—*Continued:*
roofing, 630
shoe, 553
specific-gravity test for, 1066
sundry, 613
vegetable, 499
weathering of asphalts containing, 1171
Fineness-factor of fillers, 1172
Fire coal, 312
Fireproofing processes, 45, 623, 628
Fixateur, 558
Fixed-carbon, 281
Fixed-carbon test, 949
Flammability, test for, 556
Flash-coils, 417
Flashed residue, 437
Flashings, 642
Flash-point test, 935
Cleveland, 941
of bituminous-solvent compositions, 1118
Pensky-Martens, 935
Tag closed, 945
Tag open, 948
Flash-points of solvents, 516, 948
Flat varnishes, 747
Flax felt, bituminated, 703
Flexural-strength test, 1036
Flexure, test for, 556
Float test, 820
Flocculate, 1060
Flocculation, 503
irreversible, 66
reversible, 66
Flocculative reaction, 66, 515
Floor compositions, 742
body-coat for, 742
dressing for, 742
finishing-coat for, 742
primer for, 742
Floor-coverings, 693
production of, 111
testing of, 1072
Floors, asphalt-mastic, 604
early use of asphalt for, 8, 24
Floor tiles, 20, 555, 598
Floor treads, 557
Flotation oils, 366, 370 (insert), 491, 559
Flotation process, 124, 559
Flow-point test, 917
Flow test for ship-enamel, 533
Fluidity, increasing when melted, 483
Fluorescence of tar products, 781
Flux, 99, 104, 431, 482, 570
asphalt, 428
hard, 605
paving, 570
Pittsburgh, 431, 452
roofing, 431
soft, 606
Ventura, 431

Fluxing of bituminous substances, 477, 478, 482, 491, 492
Fluxing oil, 431
Flux oil, 414, 431, 570
Foot paths, 46
Foot pavements, asphalt-mastic, 47, 604
Foots oil, 415, 416
Formaldehyde, reactions with, 59, 79, 301, 375, 376, 377, 401, 386, 497, 721, 999
Formolite reaction, 999
Formolite residue, 1000
Fossils preserved by asphalt, 2
Fossil wax, 132
Foundation course of:
  asphalt-block pavements, 597
  bituminous concrete, 583
  bituminous macadam, 587
  sheet-asphalt pavements, 590
  wood-block pavements, 602
Fraas' breaking-point test, 886
Fractional distillation of:
  bone tar, 408
  coal tar, 44, 355, 357, 363, 364
  lignite tar, 319, 320
  peat tar, 309
  petroleum, 412, 418
  shale tar, 332
  wood tar, 292, 296
Fractioning columns, 363, 418, 419, 420
Fracture, 784
France, asphalt in, 45, 124, 150, 191
  extraction with water, 124
  use in pavements, 192
Free acids, 1001
Free asphaltous acids, 1007, 1008
Free carbon, 71, 340, 376, 965, 1148, 1149
  composition of, 966
  filtration of, 969
  solubility of, 966
Free mineral matter, 170, 955
Friction elements, 552
Frosted varnishes, 747
Frothing oils, 559
Fuel lignite, 312, 313
Fuel oil from:
  petroleum, 76, 415, 416, 418, 428, 430
  shale tar, 334
Fuel-oil-gas tar, 383
Fuel, use of asphalt as, 33
Fuels, bituminous, 558
Fullers grease, 398
Fullers-grease pitch, 386, 398
Furol (Saybolt) viscosity test, 812
Fusel-oil tar, 280
Fuses, blasting, 711
Fusibility of bituminous substances, 477, 478
Fusing-point, see "softening-point"

Gagate, 34, 37, 39, 96
  Latin word for, 2
Gagat-kohle, 269
Galicia, ozokerite in, 132
Garbage pitch, 386, 397, 402, 405
Garj, 215
Gas:
  Blau, 383
  blue, 379
  carburetted water-gas, 379
  coal, 45, 339, 342
  fuel-oil, 383
  generator, 286
  illuminating, 43, 45, 339, 342, 380
  lignite, 330
  marsh, 59, 90
  Mond, 307
  natural, 59
  oil, 286
  oil-water, 382
  permanent, 281, 286, 330, 338
  Pintsch, 382
  producer, 285, 313, 316, 348
  reformed, 383
  water, 286
Gas coal, 336
Gas liquor, 370 (insert)
Gas oil from:
  lignite tar, 321
  petroleum, 279, 415, 416, 418, 422, 425, 430
  shale tar, 333, 334
Gasoline, 429
Gas pressure, effect on bitumens, 88
Gas-producer coal-tar, 348, 351
  properties of, 351, 778
Gas-producer coal-tar pitch, 63, 371
  properties of, 371, 778
Gas-producer peat-tar, 307
Gas-recirculation method of distillation, 361
Gas-works coal tar, 279, 339
  classification of, 57, 63
  coefficient of expansion of, 875
  dehydration of, 344, 350
  distillation of, 354
  from horizontal retorts, 351, 778
  from inclined retorts, 351, 778
  from vertical retorts, 351, 778
  mixtures with sludge asphalt, 464
  production of, 106
  properties of, 338, 351, 778
  recovery of, 341
  refining of, 354
  retorts used, 340
  separation of, 344, 350
  solubility of, 525 (insert)
  yield of, 338, 343
Gas-works coal-tar pitch, 371
  classification of, 58, 63

Gas-works coal-tar pitch—*Continued:*
from horizontal retorts, 371, 372, 778
from inclined retorts, 371, 372, 778
from vertical retorts, 371, 372, 778
properties of, 371, 372, 778
solubility of, 525 (insert)
Gas-works retort, 340
continuously operating, 340
horizontal, 340
inclined, 340
vertical, 340
Gel, 503
Gelatinizing of paints, 725, 727, 749, 753
Generator gas, 286
Generator tar, 289, 318
Geological formations, 82
Geology of:
bitumens, 82
pyrobitumens, 82
Geomyricite, 96
Germany:
albertite in, 269
asphalt in, 40, 197
glance pitch in, 242
Ghir, 2, 219
Gilsonite, 227
classification of, 55, 57, 62
correcting heterogeneity with, 484
discovery of, 49, 227
distillation of, 230
electrical resistance of, 543
jet, 229
metamorphized, 236
metamorphosis of, 262
mining methods, 233
mixtures with:
linseed oil, 735
paraffin wax, 484
residual oil, 229
molecular weight of, 990
nickel in, 71
production of, 102, 103
properties of, 228, 778
sampling of, 771
solubility of, 525 (insert)
weather-resistance of, 488
Gilsonite firsts, 228
Gilsonite in:
Asia, 236
North America, 230
Oregon, 235
Russia, 236
United States, 230
Utah, 49, 230
Gilsonite jet, 229
Gilsonite seconds, 228, 229
Gilsonite selects, 228, 229
Gir, 2, 219
Glance pitch, 227, 236
classification of, 55, 57, 62
Glance pitch—*Continued:*
metamorphosis of, 238, 262
properties of, 236, 778
sampling of, 771
solubility of, 525 (insert)
uranium in, 71, 240
uses in varnishes, etc., 238
vanadium in, 240, 243
weather-resistance of, 488
Glance pitch in:
Albania, 241
Barbados, 237
Central America, 241
Colombia, 241
Cuba, 238
Dead Sea, 243
Europe, 242
Germany, 242
Haiti, 238
Iraq, 242
Levant States, 242
Mesopotamia, 243
Mexico, 239
Nicaragua, 241
North America, 237
Salvador, 241
Santo Domingo, 238
South America, 241
Syria, 242
United States, 240
Utah, 240
West Indies, 237
Gloss test for bituminous-solvent compositions, 1120
Glycerides, 1002
Glycerin pitch, 408
composition of, 408
production of, 408
uses of, 408
Glycerol, 1001, 1006
Goudron, 386
Grab-test of tensile-strength, 1107
Grahamite, 227, 243
characteristics of, 244
classification of, 55, 57, 62
depolymerization of, 259
discovery of, 48, 245
fluxing of, 245, 259, 480, 492
metamorphized type of, 253
metamorphosis of, 262
mining of, 257
mixtures with residual oil, 250, 258, 1009
production of, 102, 103
properties of, 244, 778
reducing the softening-point of, 244
sampling of, 771
solubility of, 525 (insert)
types of, 248, 257
vanadium in, 71, 259, 260
weather-resistance of, 488

Grahamite in:
Argentina, 259
Colorado, 252
Cuba, 254
Mexico, 253
North America, 245
Oklahoma, 247
Peru, 260
South America, 256
Texas, 247
Trindad, 256
United States, 245
West Virginia, 48, 245
Granular matter, 499
inorganic, 499, 1055
organic, 499, 1071
Granularmetric analysis, 1056
Granules, 632, 644
cement-coated, 633
ceramic, 633
chemical, 633
coarse, 634
colored, 633
dyed, 634
metallized, 633
miscellaneous, 634
natural, 633
painted, 634
silicated, 633
sulfurized, 633
thermo-chemical, 633
uncolored, 632
Gravitation, effect on bitumens, 89
Gravity, see "specific-gravity"
Grease:
black, 394
bone, 396
penetration-test of, 836
petroleum, 431
pitch, 346, 370
still, 333
stuffing, 399
unworked consistency of, 839
waxy, 370
wool, 386, 398, 479, 1006
worked consistency of, 840
yellow, 396
Greece, asphalt in, 28, 151, 212
Greek fire, 30, 39, 40
Green rosin-oil, 303
Grooving of roofings, 1154
Grout, asphalt, 531, 570 (insert)
Grout, bituminous, 531, 1017
Grout fillers for pavements, 570 (insert), 571
Guffa, 20, 28
Guzsasphalt, 604
Gutta-percha pitch, 58

Hair cracks, 1155
Hair-felt, bituminated, 703
Haiti, glance pitch in, 238
Halogens, reactions with bituminous substances, 58, 80, 444, 453
Halowax, 542, 543
Hardening, bituminous substances, 80, 301, 376, 377, 402, 485
Hardening, spontaneous, 1150, 1154
Hard flux, 606
Hardness of bituminous substances, 477, 478
Hardness scale, 829
Hardness tests, 828
consistometer method, 840
of bituminized aggregates, 1019
of bituminous-solvent compositions, 1120
Moh's scale, 829
needle-penetrometer method, 830
Hard wax, 132
Hardwood distillation, 279, 290, 293
Hardwood tar, 297
classification of, 57, 63
properties of, 298, 778
solubility of, 525 (insert)
Hardwood-tar pitch, 299
classification of, 57, 63
composition of, 300
properties of, 299, 778
solubility of, 525 (insert)
Hartine, 96
Hartite, 96
Hatchettine, 46, 135
Hatchettite, 46, 135
Heat:
effect on asphaltic pyrobitumens, 72, 73
effect on bitumens, 89
effect on bituminized aggregates, 1037, 1038
effect on bituminous substances, 72, 89
effect on fatty-acid pitches, 401
resistance of bituminized fabrics to, 1085
resistance of bituminous-solvent compositions to, 1121
use of molten metals to, 360, 454, 594
Heat content, test of, 870
Heating of:
asphalts, 115
bitumens, 115
bituminous substances, 115, 491
native asphalts, 115
pyrobitumens, 115
Heat tests, 777, 863
Heavy naphtha, 365, 366
Heavy oils from:
coal tar, 346, 356, 364, 366, 370 (insert)
peat tar, 308, 309
shale tar, 466
wood, 292, 295, 296
Henderson retort, 329
Hiding test for bituminous-solvent compositions, 1120
High-temperature carbonization, 338

High-temperature coal tar, distinguishing, 350
Historical review, 1
Hofmannite, 96
Holzzement, 375, 527
Homogeneity tests, 483, 781, 1133
  in solution, 782
  to the eye, 782
  under microscope, 782, 1133
  when melted, 782
Homogenizer, 505, 506
Horizontal retort, 340, 345
Hungary, asphalt in, 204
  extraction with water, 204
  use for mastic, 204
  use in paving, 204
  use in rubber goods, 204
Husbandry, early use of asphalt in, 38
Hutchinson viscosity test, 819
Hydraulic main, 341, 343, 382, 383
Hydrocarbons:
  aliphatic, 281, 728
  aromatic, 281, 283, 337, 381, 728
  cyclic, 349, 409
  naphthene, 336, 337, 349, 409
  olefine, 68, 337
  open chain, 409
  paraffin, 68, 337, 349, 409
  polycyclic, 990, 1011
  saturated, 65, 349, 995
  unsaturated, 65, 337, 349, 995
Hydrogen, 65, 68, 74
  determination of, 982
  reactions with bituminous substances, 58, 74, 486
Hydrogenated products, 58
Hydrogenation, 74, 429, 486
Hydrolene, 446
Hydrometer method of specific-gravity, 790
Hydrophile, 504
Hydrophobe, 504
Hydrosol, 503
Hydrostatic pressure, effect on bitumens, 88

Ichthyol, 203
Identification, see "analysis of"
Illuminating gas:
  discovery of, 43
  enriching of, 380
  manufacture of, 339, 343, 380
  yield of, 343
Illuminating oil from:
  lignite tar, 320, 321
  petroleum, 415, 416, 430
  shale tar, 333, 334
Imitation leather, 709
Immediate pitches, 58
Immersion test, see "absorption test"
Impact tests:
  cantilever-beam method, 1041
  Charpy, 1040
Impact tests—*Continued:*
  Izod, 1041
  on molded compositions, 556, 1039
  on paving compositions, 1022
  simple-beam method, 1040
Impregnated rock, 86
Impregnating compounds, 491, 493, 542
Impregnation accelerators, 483
Impregnation of:
  acoustical blocks, 719
  acoustical felt, 714
  armature windings, 542
  asbestos-cement panels, 546
  belting, 546
  bibulous bodies, 546, 554
  brake-linings, 552, 713
  building blocks, 546
  clay articles, 546
  clutch-facings, 552, 713
  compressed wood-pulp, 546
  concrete, 546
  conduits, 546, 713
  cords, 712
  coverings for wires, 712
  ebonized lumber, 546
  felt, 634
  flashings, 642
  flooring, 693
  insulating board, 718
  insulating tape, 715
  magnet coils, 542
  membrane waterproofing, 697
  paper, 706, 707
  pipes, 546, 557
  plaster-board, 719
  roofing felt, 368, 625, 642
  roofings, 625, 634
  ropes, 712
  shingles, 625, 634
  stone, 546
  storage-battery boxes, 546
  stucco-base, 719
  tacking-strips, 714
  transformer coils, 542, 546
  tubes, 546
  wall-board, 717
  waterproofing fabrics, 642, 698
  wood-blocks, 598
Impsonite, 270
  classification of, 55, 57, 62
  copper in, 272
  depolymerization of, 72
  metamorphosis of, 95, 262, 270
  properties of, 262, 270, 778
  vanadium in, 71, 273
Impsonite in:
  Arkansas, 271
  Australia, 274
  Brazil, 273
  Mexico, 253
  Michigan, 272

Impsonite in—*Continued:*
Nevada, 272
North America, 271
Oklahoma, 271
Peru, 273
South America, 272
United States, 271
Incas, use of asphalt by, 41
Inclined retort, 340, 341
Indentation test, 555
India, asphalt in, 16, 37, 221
Indiana, asphalt in, 159
Indians, use of asphalt by, 15
Individual particle, 1060
Individual shingles, 660
Inorganic aggregate, see "mineral aggregate"
Inorganic theories of petroleum origin, 89
Insoles, 553
Insoluble in:
benzol, 965
carbon disulfide, 951
carbon tetrachloride, 962
petroleum naphtha, 964
sulfuric acid, 995
various solvents, 970
Insulating board, 718
Insulating compounds, electrical, 368, 491, 493, 541, 542, 547, 550
Insulating fabrics, 709, 714
Insulating felts, 714
Insulating japans, 753, 754
Insulating papers, 703, 709
Insulating tape, 714, 1086
Insulation:
adhesion test of, 1087
cold, 552, 703, 714, 718
electrical compositions, 541, 542, 547, 550
electrical fabrics, 709, 712, 713, 714
electrical japans, 753, 754
for wires, 712
heat, 703, 714, 718
oven test of, 1087
sound, 714
tackiness test of, 1087
tensile-strength test of, 1086
vibration, 714
Integral waterproofing:
compounds, 560
methods, 564, 696, 704
Interfacial-tension, 504, 862
Intermediate oil, 329, 466
Intermittent distillation of:
asphaltites, 230
asphalts, 39, 40, 119, 203, 204, 209, 280
bones, 407
bone tar, 408
coal, 340, 345, 379
coal tar, 355, 357
coal-tar pitch, 369
Intermittent distillation of—*Continued:*
fatty acids, 389, 395
hardwood, 290
lignite tar, 319
ozokerite, 130
peat, 307
peat tar, 309
petroleum, 412, 414
rosin, 301, 302
shale tar, 332
soft wood, 295
wood, 292, 296
wurtzilite, 472
Internal phase, 503, 504
Introfiers, 483
Iodine number, 80
Iodine, reactions with bituminous substances, 58, 80
Ionite, 96
Iraq (Irak):
asphalt in, 5, 21, 38, 152, 219
glance pitch in, 243
Iron pitch, 190
Italy, asphalt in, 124, 206
distillation of, 280
extraction with water, 124
use for paving, 208
Izod impact-test, 1041

Japan, asphalt in, 221
Japans, 753
baking, 753
electrical insulating, 753, 754
emulsion, 755
mat, 756
sampling of, 775
water, 755
wrinkle-finish, 755
Japan wax, 543
Jelly, asphalt, 566
Jelly, petroleum, 431
Jet, 96
Latin word for, 2
Jet gilsonite, 229
Jir, 219
Joint-filler, 570 (insert), 594
Joints, bituminous expansion, 491, 493, 611
armored, 615
laminated, 614
preformed, 551
premolded, 612
reinforced, 614
Jugoslavia, asphalt in, 202
Junction-box compound, 546

Kabaite, 135
Kapak, 472
Karasakiz, 219
Karbolpech, 377

Katran, 215
Kaumazite, 316
Kentucky, asphalt in, 139, 155
  use in pavements, 158
Kerites, 266
Kerogen, 277, 485
Kerosene, 430
Kerosene test for felt, 1110
Kerotenes, 266
Kerzenteer, 386
Kettle, varnish, 747
Kimmeridge shales, 331
Kindebal, 132
Kinderball, 132
Kir, 2, 59, 215
Kitt, 1
Knife breaking-point test, 886
Köflachite, 96
Korite, 446
Kramer-Sarnow softening-point test, 898
Kresolharz, 377
Kukkersite shale, 331, 335
Kupru, 2, 26

Lacquers, bituminous, 720
Lacquers, cellulose-ester, 751
Lactones, 1002
Lactone value, 1002
Lake Asphaltites, 30, 36
Lake-dwellers, use of asphalt by, 27
Lakes of asphalt, 84, 146, 152, 182, 190
Lake, Trinidad, 93, 182
Laminated expansion-joints, 614
Laminated roofings, 653
Laminated shingles, 660
Land asphalt, 189
Lap-cement for roofings, 683, 734
Latex, 487, 513, 613, 629, 694, 707, 712
Laying roofings, 685
Laying shingles, 685, 688
Leather, bituminated, 557
Leather, imitation, 709
Leather tar, 280
Leather-tar pitch, 280
Lep, 132, 134
Leucopetrin, 96
Leucopetrite, 96
Levant States, asphalt in, 152, 216
  glance pitch in, 242
Leyteite, 154
Libollite, 270
Liebermann-Storch reaction, 1016
Light, effect on asphalt, 1011, 1147
Lighting, early use of asphalt for, 31
Light oil from:
  coal tar, 346, 356, 365, 370 (table)
  peat, 308, 309
  wood, 292, 295, 296
Lignin tar, 280
Lignin-tar pitch, 280
Lignite, 96
  bituminous, 312
  briquetting of, 313, 314, 317
  classification of, 55, 57, 62
  distillation of, 279, 313, 316
  extraction with solvents, 313, 314
  formation of, 312
  fuel, 312, 313
  geology of, 83
  impure, 315
  Latin word for, 2
  low-temperature distillation of, 316
  metamorphosis of, 94, 96
  mining of, 311, 312
  non-bituminous, 312
  properties of, 778
  retort, 312, 313, 314
  varieties of, 310
  volatile matter in, 282
  yield on distillation, 315
Lignite creosote, 321
Lignite paraffin, 320
Lignite shales, 276
  classification of, 55
  properties of, 778
Lignite tar, 279, 310, 321
  blowing of, 323
  classification of, 57, 63
  coke-oven, 317
  composition of, 317, 318
  cracking of, 429
  dehydration of, 315, 317
  discovery of, 44
  distillation of, 319, 320
  hydrogenation of, 77
  low-temperature, 317
  producer-gas, 317
  production of, 107, 279
  properties of, 317, 318, 778
  refining of, 318, 320, 321
  retort, 317
  solubility of, 525 (insert)
  yield of, 316
  yields on distillation, 320
Lignite-tar pitch, 310, 320
  blowing of, 323
  classification of, 58, 63
  distinguishing, 323
  fluxing of, 479
  from gas-producers, 285
  hardening of, 322, 323
  production of, 107, 279
  properties of, 322, 479, 778
  solubility of, 323, 525 (insert)
  sulfurized, 323
  weather-resistance of, 479
Lignite-tar-sludge pitch, 320, 321
Lignitic residues, 97
Lignitic shales, 62, 94, 276, 277
Limestone, asphaltic, 56

Limmer asphalt, 43, 197, 604
Linoleum tar, 280
Linoleum-tar pitch, 280
Linoxyn, 735, 740
Liquefying-point test, 917
  relation to solidifying-point, 892
Liquid asphalt, 138, 431
Liquid paraffin, 431
Liquid sulfur dioxide, reactions with bituminous substances, 77
Litho-carbon, 170
Lobsann asphalt, 196
Long residuum, 424
Lothian shales, 331
Louisiana, asphalt in, 169
Love-potions, use of asphalt in, 39
Low-temperature carbonization, 338
Low-temperature coal tar, 348
  coefficient of expansion of, 875
  composition of, 338, 349
  cracking of, 429
  distillation of, 354
  distinguishing from high-temperature tar, 350
  hydrogenation of, 77
  properties of, 351, 778
  solubility of, 525 (insert)
  yields of, 338, 348, 349
Low-temperature coal-tar pitch, 371
  coefficient of expansion of, 875
  properties of, 371, 778
  yields of, 349
Low-temperature lignite-tar, 317
Lubricating oil, 430
  from lignite-tar, 320, 321
  from peat-tar, 309
  from petroleum, 76, 126, 415, 416, 418, 430
  from shale-tar, 333, 334
  propane refining of, 126
Lubricating stock, 423, 424
Lumber:
  artificial, 557
  ebonized, 546
Lustre, 785
Lyophile, 504
Lyophilic portion of asphalts, 66
Lyophobe, 504
Lyophobe sol, 66
Lyophobic portion of asphalts, 66

Macadam, bituminous, 583
Madagascar, asphalt in, 226
Magma oil, 398
Magnet coils, 542
Maltha, 35, 44, 59
  Greek word for, 2, 59
  Latin word for, 2
Malthenes, 66, 964
Mandrel test for pliability, 1080
Manitoba, asphalt in, 180
Manjak, 227, 237, 256
Manufactured products, 476
Maracaibo asphalt, 149
Marble wax, 132
Marine paints, 740, 741
Marsh gas, 59, 90
Masonry coatings, 731
Mastic, acid-proof, 604, 605
Mastic asphalt, 45, 104, 204
  blocks of 605
  early uses of, 8, 15, 16, 20, 22
  word in:
    Arabic, 2
    Sumerian, 2
    Turkish, 2
Masticator, 506
Mastic cake, 104, 201, 605
Mastic floors, 604
Mastic mortar, 531
Mastic pavements, 46, 604
Mastic roofs, 609
Mastic, rock-asphalt, 45, 605
Mastix, 1
Masut, 431
Matka, 132
Mazout, 431
Mazut, 431
Mechanical analysis of aggregates, 1056
Mechanical tests, 809
Medicine, uses of asphalt in, 18, 30, 35, 37, 38, 39
Medium, 66
Melanchyme, 96
Mellite, 96
Melting-point, see "softening-point"
Melting-point test for:
  paraffin wax, 914
  petrolatum, 913
Melting tanks, 491
Membrane waterproofing, 639, 696, 700, 704
Membrane-and-plastic waterproofing, 704
Mercuration, degree of, 1000
Mesopotamia:
  asphalt in, 5, 24, 27, 33, 152, 219
  glance pitch in, 243
Messel lignite, 285, 311, 316
Metallic salts, reactions of, 58, 81, 497, 808
Metalloids, reactions of, 79
Metamorphosis of:
  albertite, 95, 262
  anthracite coal, 94, 97
  asphalt, 93
  asphaltic pyrobitumen, 93, 94
  asphaltic pyrobituminous shales, 94
  asphaltites, 93, 94
  bitumens, 89
  bituminous coal, 94, 97
  cannel coal, 96

Metamorphosis of—*Continued:*
cellulose, 94
coal shales, 94
elaterite, 95, 262
impsonite, 95, 262, 270
lignite, 94, 96
lignite shales, 94
mineral waxes, 93, 94
native asphalts, 94
non-asphaltic pyrobitumens, 95
ozokerite, 94
peat, 94, 96, 306
petroleum, 94
wurtzilite, 95, 262
Methods of examination, see "analysis"
Mexico:
asphalt in, 143, 180
extraction with water, 123
use as chewing-gum, 49
glance pitch in, 239
grahamite in, 253
impsonite in, 253
Micelle, 66, 809
Michigan, impsonite in, 272
Micro-asbestos, 498, 538
Microns, 803
Microscopic examination of:
bituminous dispersions, 1133
bituminous substances, 187, 480, 782, 802
colloidal particles, 802, 1062
fibers, 1111
mineral matter, 802, 1062
Middle oil, 346, 356, 365, 366, 370 (insert)
Middletonite, 96
Mildewing, prevention of, 538, 640, 698
Mill:
colloidal, 505, 506, 507
paint, 731
Mineral aggregate:
air-separation test for, 1060
bituminized, 567, 1017
classes of, 574, 576
coarse, 500, 574 (insert)
combinations of, 500
definition of, 574, 1060
dense, 582, 583
elutriation test of, 1058
examination of, 1055
fine, 574 (insert)
graded type of, 582, 583
grading of, 592
granularmetric analysis of, 574 (insert), 1056
incorporation of, 494
intermediate type of, 574 (insert)
macadam type of, 581, 583
open type of, 581, 583
percent of wear of, 575
separation from bituminous matter, 1044
sieve tests of, 574 (insert), 1056
Mineral aggregate—*Continued:*
specific-gravity of, 1066
toughness of, 575
types of, 574 (insert), 576
voids in, 1071
weight per cubic foot, 575
Mineral aggregate, use in:
asphalt blocks, 596
asphalt mastic, 606, 609, 611
bituminous concrete, 576, 587, 588
bituminous macadam, 576, 583, 584
pavements, 576
sheet-asphalt pavements, 576, 590, 591, 592
surface treatment of pavements, 579, 580, 581
Mineral fat, 132
Mineral fillers, 488, 497, 500, 501
adsorptive properties of, 525, 1066
compacting weight of, 1172
drying of, 496
effect on physical properties of asphalts, 500, 1175
effect on physical properties of coal-tar pitch, 501
effect on weather-resistance of asphalts, 489, 1154, 1171
examination of, 1055, 1128
fineness-factor of, 1172
for use in:
paving compositions, 574 (insert)
roofings, 630
solvent-compositions, 724
graded, 488
incorporation of, 494
microscopic examination of, 1062
physical properties of, 1176
preheating of, 496
specific-gravity of, 1066
Mineral matter:
air-separation test for, 1060
associated with asphalts, 70, 155
chemical analysis of, 955, 1129
combined with non-mineral constituents, 70, 957
elutriation test of, 1058
free, 70, 955
granularmetric analysis of, 574 (insert), 1056
microscopic examination of, 802, 1062
separation from bituminous constituents, 1044
separation from fibrous constituents, 1099
sieve test for, 1056
specific-gravity of, 1066
surfacings of, 632, 644
voids in, 632, 800, 1017, 1071
uncombined, 70, 955
Mineral oil, see "petroleum"

Mineral pitch, 44, 221
Mineral rubber, 547
Mineral spirits, 430
Mineral surfacings for roofings, 1099
Mineral tar, 59
Mineral wax, 129
  classification of, 55, 62
  composition of, 54
  definition of, 55
  effect of heat on, 73
  fluxing of, 478
  geology of, 82
  metamorphosis of, 93
  origin of, 54
  properties of, 54, 478, 778
  solubility of, 525 (insert), 729
  weather-resistance of, 478
  word in:
    Arabic, 2
    Greek, 2
    Latin, 34
    Turkish, 2
Mining methods, 112
  open-cut quarrying, 112, 233
  tunnelling, 112, 324
  special methods, 112
Missouri, asphalt in, 159
Mixed-base petroleum, see "semi-asphaltic petroleum"
Mixed-in-place pavements, 583
Mixers, 491, 494, 496, 554, 555
Mixing type of bituminous dispersion, 1138
Mixtures of bituminous substances, 477, 481
  binary, 481
  complex, 489
  tertiary, 489
Mobility, definition of, 828
Mobility test for bituminous-solvent compositions, 1118
Modified accelerated weathering test, 1177
Moh's hardness scale, 829
Moisture, see "water"
Moisture, effect on:
  adhesion, 1018
  bituminized fabrics, 1084
  bituminized mineral aggregates, 1018, 1029
  bituminous dispersions, 1139
  bituminous-solvent compositions, 1121
  bituminous substances, 785, 1150
  molded compositions, 1029
  paving compositions, 1018
Molasses tar, 280
Molasses-tar pitch, 280
Molded compositions, 491, 550, 1025
  analysis of, 1044
  compressive strength test of, 1034
  distortion under heat of, 1037
  electrical tests of, 1044
Molded compositions—*Continued:*
  flexural strength test of, 1036
  resistance to impact, 1039
  resistance to moisture, 1029
  separation into component parts, 1044
  softening-point test of, 1038
  tensile-strength of, 1033
  thickness test of, 1025
Molded shingles, 554
Molding compounds, 104
Molding of bituminous substances, 550
Molecular weight, 400
  test for, 990
Molybdenum in asphalts, 71
Mond tar, 307, 350
Montan pitch, 58, 137
  electrical resistance of, 543
  solubility of, 525 (insert)
Montan-tar pitch, 280
Montan wax, 132, 136
  blown, 137
  characteristics of, 137
  chlorinated, 137, 721
  classification of, 56, 62
  composition of, 136
  distillation of, 137
  electrical resistance of, 543
  extraction with solvents, 128, 136, 313, 314
  fluxing of, 478
  from lignite, 136
  from pyropissite, 136
  in Saxony, 136
  mixtures with bituminous substances, 489
  properties of, 478, 778
  raising the softening-point of, 137
  refined, 137
  saponification of, 1003
  solubility of, 525 (insert)
  weather-resistance of, 478
Montan-wax pitch, 137
Montan-wax tar, 280
Mortar, early use of asphalt for, 8, 12, 20 21, 23, 25, 26, 28, 29, 31, 33, 37, 151
Mound tar, 289
M. R. (mineral rubber), 547
Muckite, 96
Mulching paper, 710
Mullen strength of:
  bituminized fabrics, 1078
  felt, 1104
  paper, 1104
Multiple shingle-strip, see "shingle-strip"
Mûmia, 18, 37
Mummies, 17, 18, 32, 37

Nails for roofings, 683
Nails for shingles, 683
Naphatil, 132
Naphtha, heavy, 365, 366
  solvent, 365

Naphthalene, 283, 342, 346, 366, 381, 483
determination of, 992
Naphthalene oil, 364, 366
Naphthene hydrocarbons, 336, 337, 349, 409
Naphthol pitch, 377
classification of, 58
hardening of, 377, 486
identification of, 377
reaction with formaldehyde, 486
solubility of, 377, 525 (insert)
toughening of, 377
uses of, 377
Naphthylamine pitch, 281, 377
Native asphalt, 139, 155
asphaltic constituents in, 1009
associated with mineral matter, 155
behavior on heating, 72, 89
blown, 440
classification of, 55, 56, 62
comminution of, 121, 158, 169, 201, 499
dehydration of, 114
distillation of, 119, 280
distinguishing from residual asphalts, 456, 457
extraction of:
with solvents, 125
with water, 122, 124, 165, 177, 178
fluxing of, 478, 492
heating of, 115, 492
impure, 155
metamorphosis of, 94
mining methods, 112
mixtures with coal tar and coal-tar pitch, 484
nitrated, 78, 486, 726
presence of iron and aluminium in, 70
production of, 100, 102, 103
properties of, 478, 778
pure, 139
refining of, 114
sedimentation of, 122
shipping of, 113
solubility of, 525 (insert)
sulfurized, 452
transportation of, 113
weather-resistance of, 478
word in:
Accadian, 2
Arabic, 2, 219
Assyrian, 2
Greek, 2
Hebrew, 2
Latin, 2
Russian, 219
Sanskrit, 2
Spanish, 59
Sumerian, 2
Turkish, 2, 219
Native mineral waxes, see "mineral waxes"
Natural asphalts, see "native asphalt"
Natural gas, 59
Needle penetrometer, 830
Neft-gil, 2, 96, 132
Nephtgil, 132
Neuchatel asphalt, 43, 195
Neudorfite, 96
Neutral fats, 1002
Neutral oil, 415, 416
Nevada, impsonite in, 272
New York, discovery of bituminous matter in, 47
Nicaragua, glance pitch in, 241
Nickel in asphalts and asphaltites, 16, 71
Nigeria, asphalt in, 226
Nigrite, 262, 269
Nitrated asphalt, 78, 486, 726
Nitrated asphaltite, 726, 752
Nitrated coal-tar, 721, 739
Nitrated coal-tar pitch, 739
Nitric acid, reactions with, 78, 401, 486
Nitro-cellulose, see "cellulose-ester lacquers"
Nintrogen, 69, 92, 346
determination of, 988
Nomenclature, see "definitions"
Non-asphaltic petroleum, 55, 62, 72, 73, 409, 410, 778
classification of, 55
Non-asphaltic pyrobitumens:
behavior on subjecting to heat, 73
classification of, 55, 62
composition of, 54
definition of, 57
effect of heat on, 73
geology of, 82, 83
metamorphosis of, 95
origin of, 54, 95
properties of, 54, 778
solubility of, 54
veins of, 86
Non-asphaltic pyrobituminous shales, 276
classification of, 55, 57
Non-bituminous lignite, 312
Non-bituminous substances, properties of, 479
Non-coking coal, 345
Non-mineral matter insoluble in carbon disulfide, 480
Non-uniformity, 1061
North America:
albertite in, 267
asphalt in, 139, 155
gilsonite in, 230
glance pitch in, 237
grahamite in, 245
impsonite in, 271
ozokerite in, 134
wurtzilite in, 264
Nova Scotia, albertite in, 48

"Number" of:
burlap, 625, 1103
duck, 625, 1103
felt, 622, 1102
paper, 704, 1100
woven fabrics, 1103

Obispo, 446
Oil or oils:
acetone, 292
acid, 294, 415
acid-distillate, 415, 460
acid-naphthalene, 366
animal, 479, 482, 722, 723
anthracene, 346, 364, 366
asphaltic, 55, 62, 409, 410
asphaltum, 431
black, 431
blue, 334
blue rosin, 303
bone, 407
burning, 76
carbolic, 356, 370 (insert)
carburetting, 380
coal-tar distillate, 601
coal-tar paving, 601
collecting, 559
creosote, see "creosote oil"
cylinder, 415
dead, 366
dégras, 399
Dippel, 407, 1147
distillate, 600
dust-laying, 431, 575
enriching, 380
flotation, 366, 370 (insert), 491, 559
flux, 414, 431, 570
fluxing, 431
foots, 415, 416
frothing, 559
fuel, see "fuel oil"
gas, see "gas oil"
green, 332
heavy, see "heavy oil"
illuminating, see "illuminating oil"
intermediate, 329, 466
light, see "light oil"
lubricating, see "lubricating oil"
magma, 398
middle, 346, 356, 365, 366, 379 (insert)
naphthalene, 364
neutral, 415, 416
once-run, 332
paraffin, 320, 321, 334, 416, 417
pine, 296
pitch, 346
red, 320, 321
residual, 414, 432
residuum, 414

Oil or oils—*Continued:*
road, 104, 431
rosin, 296
rubber, 483
seek, 298
shale, see "shale tar"
tar, 290
tar-acid, 366
vaseline, 320
vegetable, see "vegetable fats and oils"
wood, 289, 292
wool, 397, 398
yellow rosin, 302
Oil-bearing shales, 275
Oil-forming shales, 275
Oil gas, 286
Oil-gas tar, 279, 286, 287, 378, 382
classification of, 57, 63
high-temperature, 384, 778
low-temperature, 384, 778
production of, 106
properties of, 383, 384, 778
refining of, 383
solubility of, 525 (insert)
sulfurized, 383
Oil-gas-tar pitch, 378
characteristics of, 385
classification of, 58, 63
distinguishing from:
coal-tar pitch, 384
water-gas-tar pitch, 384
fluxing of, 479
properties of, 384, 385, 479, 778
resistance to moisture, 385
solubility of, 385, 525 (insert)
weather-resistance of, 479
Oil-penetration test for felt, 1109
Oil-resistance test, 1122
Oil shales, 275, 485
Oil-water gas, 382
Oil-water-gas tar, 383, 384
Oily constituents in asphalts, 66, 457, 1008, 1012
Oily medium of asphalts, 67
Ointments, use of asphalt in, 36, 44
Oklahoma:
asphalt in, 140, 159
grahamite in, 247
impsonite in, 271
Oklahoma asphalt:
blown, 166
extraction with water, 123, 165
resistance to oxidation, 167
use in pavements, 165
Okonite, 130
Olefines, 68, 337
Oleoresins, 301, 304
Oleosol, 503
Oleum animale foetidum, 407
Open-chain hydrocarbons, 409

Oregon:
asphalt in, 143
gilsonite in, 235
Orepuki shale, 331
Origin of:
asphalt, 89
asphaltic pyrobitumens, 89
bitumen, 89
non-asphaltic pyrobitumens, 95
petroleum, 54, 89
pyrobitumens, 54, 82
Ornamental roofings, see "roofings"
Ornaments, early use of asphalt for, 5, 7, 11, 12, 13
Ostatki, 431
Oven for volatility test, 920
Oven tar, 289
Oven test for tape, 1087
Overheating of bituminous substances, 492
Oxidation of bituminous substances, 1148
Oxidized asphalt, 49, 50, 431
Oxidized coal-tar, 49, 374, 440
Oxidized petroleum asphalt, see "blown petroleum asphalt"
Oxygen, 69
determination of, 989
in fuel, 282
reactions with bituminous substances, 58 74, 444
Oxygenated asphalt, 431
Oxygenation of asphalt, 74
Oxygenized asphalt, 431
Ozokerit, 132
Ozokerite, 129
adulteration of, 132
characteristics of, 131
classification of, 56, 62
composition of, 129
discovery of, 46
distillation of, 130
electrical resistance of, 543
extraction with water, 124, 130, 134
fluxing of, 478
metamorphosis of, 94
mining of, 130
molecular weight of, 995
occurrence of, 129
production of, 102, 103
properties of, 131, 478, 778
refining of, 130
refractive index of, 995
solubility of, 525 (insert)
weather-resistance of, 478
word in Greek, 46
Ozokerite in:
Asia, 134
Europe, 132
Galicia, 132
North America, 134
Philippine Islands, 134
Ozokerite in—*Continued:*
Poland, 132
Rumania, 133
Russia, 133
Siberia, 134
Texas, 135
Turkestan, 134
United States, 134
Utah, 134
Ozokerite pitch, 58, 130
Ozone, 58, 301, 444, 489, 755

Packing-house pitch, 386, 402
Packing-papers, 46, 709
Pacura, 431
Paint-films, analysis of, 1131
Paints, see "bituminous-solvent compositions"
Palliatives, dust, 575
Palm-oil pitch, 386, 402, 405
Paper or papers, 705, 710
air-resistance of, 1107
analysis of, 1072
building, 703
cement-curing, 703
corrugated, 710
creped, 710
duplexed, 709
electrical insulating, 709
examination of, 1100, 1104, 1111
fiber composition test for, 1111
insulating, 703
moisture content of, 1100
mulching, 710
"number" of, 704, 1100
packing, 46, 709
paraffined, 707
porosity test for, 1107
reinforced, 710
sampling of, 773
saturating speed of, 1109
sheating, 703
strength of, 706, 1077, 1104
thickness test for, 1103
weight test for, 1101
wrapping, 709
Paracumarone resin, see "coumarone resin"
Paraffin, see "paraffin wax"
Paraffinaceous mass, 466
Paraffinaceous petroleum, see "non-asphaltic petroleum"
Paraffin, determination of, 992
Paraffin distillate, 415, 416, 417, 422, 466
Paraffin hydrocarbons, 68, 337, 349, 409
Paraffin, liquid, 431
Paraffin scale, 431
Paraffin, solid, 381, 630
determination of, 992
Paraffin wax, 431, 466
chlorinated, 542, 543

Paraffin wax—*Continued:*
classification of, 56, 63
discovery of, 46
effect of, 630
electrical resistance of, 542, 543
from asphaltic petroleum, 415
from boghead-coal tar, 336
from cannel-coal tar, 336
from lignite tar, 63, 314, 318, 320, 321, 466
from non-asphaltic petroleum, 416, 417
from peat tar, 63, 308, 309, 466
from petroleum, 63, 416, 417, 431, 466
from semi-asphaltic petroleum, 416, 417
from shale tar, 63, 333, 334, 466
from torbanite tar, 336
hydrogenation of, 77
mixtures with gilsonite, 484
molecular weight of, 995
origin of name, 18, 46
oxidized, 440
properties of, 466, 467, 778
refined, 431, 466
refractive index of, 782, 995
resistance to chemicals, 467
resistance to water, 467
softening-point test for, 908, 914
solubility of, 525 (insert)
weather-resistance of, 467
Parianite, 187
Parolite, 446
Particles, size of, 803, 1056, 1060
Paulins, 639
Pavements, 567
Amiesite, 586
asphalt, 567
asphalt-block, 570 (insert), 595
asphalt broken-stone, 583
asphalt fillers for, 574 (insert), 595, 603
asphaltic binders for, 471, 570 (insert), 571
asphaltic concrete, 570 (insert), 571
asphalt macadam, 570 (insert), 571, 583
asphalt mastic, 46, 104, 604, 610
base course of, 583, 587
bedding course of, 597
binder course of, 590
binders for, 368, 431, 567, 579 (insert), 571
liquid, asphaltic, 568, 569
liquid, coal-tar, 570 (insert)
semi-solid to solid, asphaltic, 570 (insert)
semi-solid to solid, coal-tar, 570 (insert)
Bitosan, 587
Bitoslag, 587
Bitulithic, 586
bituminous broken-stone, 583
Pavements—*Continued:*
bituminous concrete, 570 (insert), 575, 586
bituminous emulsions for, 512, 572, 575, 584, 589
bituminous gravel, 583
bituminous macadam, 570 (insert), 575, 583
bituminous surfacings, 578
black base of, 587
carpet-coat of, 578
classes of, 567
coal-tar pitch binders for, 368
cold application of, 158, 169, 586
Colprovia, 587
compressed asphalt, 589
cushion layer of, 602
dispersions for, 572
dust-laying compositions for, 431, 570 (insert), 575
early use of asphalt for, 22
examination of, 1017
Filbertine, 587
first use in England, 46, 47
first use in France, 48
first use in London, 46, 48
first use in Paris, 46, 48
first use in United States, 47, 48, 49
foot, 46, 47
foundation course of, 583, 587, 590
grout filler for brick, 570 (insert), 571
grout filler for stone-block, 570 (insert), 571
joint fillers for, 570 (insert)
mastic, 46, 104, 604, 610
mineral aggregates for, 574
mineral fillers for, 574 (insert)
mixed-in-place, 583
mixture of asphalt and coal-tar pitch for, 484
paint-coat for, 602
penetration-mix for, 583
plant-mix surfacing for, 583
production of, 107
repair work and patching of, 570 (insert)
re-use of, 594
road-mix, 583
road-mix surfacing for, 581
rock asphalt, 590
sand asphalt, 590
seal-coat for, 431, 578, 583
sheet-asphalt, 570 (insert), 575, 589
sheet-asphalt topping, 589
Sintex, 587
stone-filled sheet-asphalt, 586, 589
surface course of, 578, 584, 588, 590
surface mixture for, 589
surface treatment of, 570 (insert), 579
surfacings for, 570 (insert), 579, 589

Pavements—*Continued:*
tar-concrete, 570 (insert)
tar-macadam, 570 (insert)
Topeka, 587
Trinidad asphalt, 49
Warrenite, 587
wearing-course of, 581, 583
wood-block, 47, 570 (insert), 598
Paving asphalt, 104
Paving blocks, 570 (insert), 571, 595
press for, 596
Paving compositions, tests for:
brittleness, 1024
coefficient of wear, 1025
effect of water on adhesion, 1018
examination of, 1017
extrusion of, 1022
hardness, 1019
resistance to displacement, 1020
resistance to impact, 1022
resistance to moisture, 1018
sampling of, 773
separation into component parts, 1044
shattering, 1024
specific-gravity, 1017
stability, 593
voids in, 1017
Paving materials, 491, 493
sampling of, 773, 774
Peat, 96, 305
action of heat on, 279
briquetting of, 307
classification of, 55, 57, 62
collecting of, 306
composition of, 306
dehydrating of, 307
distillation of, 279, 307
formation of, 305
geology of, 83
metamorphosis of, 94, 96, 306
properties of, 778
varieties of, 305
volatile matter in, 282
Peat paraffin, 308, 309, 466
Peat tar, 279, 305
classification of, 57, 63
composition of, 308
cracking of, 429
distillation of, 309, 310
fluxing of, 479
from gas-producers, 285, 307
producer, 308
production of, 279, 308
properties of, 310, 778
refining of, 308
retort, 308
solubility of, 525 (insert)
yields of, 308
Peat-tar pitch, 305
classification of, 58, 63
Peat-tar pitch—*Continued:*
properties of, 310, 479, 778
recovery of, 310
solubility of, 525 (insert)
weather-resistance of, 310, 479
Pedesis, 81
Penetration, definition of, 833
Penetration of bituminous substances, 477, 478
Penetration-mix pavements, 583
Penetration test, 830
Penetration type of bituminous dispersions, 1138
Penetrometer, 830
Pensky-Martens flash-point test, 935
Peptization, 503
Peptizing agent, 503
Peptizing reaction, 66, 515
Persia, asphalt in, 5, 15, 28, 33, 37, 43
Peru:
early use of asphalt in, 41
grahamite in, 260
impsonite in, 272
Petrolatum, 430
penetration test for, 836
softening-point test for, 913
Petrolenes, 47, 66, 964
Petroleum, 409
asphalt content of, 410
asphaltic, 55, 62, 72, 73, 409, 410, 778
asphalt yields from, 411
behavior on subjecting to heat, 73
classification of, 55, 62, 409
composition of, 54, 409
continuous distillation of, 417
cracking of, 278, 279, 286, 417, 424
definition of, 55
dehydration of, 411, 418
destructive distillation of, 279, 412, 414, 41(
distillates from, 429
liquid, 429
semi-solid, 430
solid, 430
distillation of, 412
dry-distillation of, 412, 413, 416
non-asphaltic oils, 414, 416
semi-asphaltic oils, 414, 416
effect of heat on, 73, 279
fluxing of, 478
fractional distillation of, 412, 418
geology of, 82
heating of, 73, 279
intermittent distillation of, 412, 414
metamorphosis of, 94
non-asphaltic, 55, 62, 409, 410, 778
occurrences throughout world, 410
origin of, 54, 89
oxidizing of, 440
paraffinaceous, see "non-asphaltic"
polymerization of, 72

Petroleum—*Continued:*
pools of, 86
propane refining of, 126
properties of, 54, 478, 778
refining of, 412
residues of, 431
semi-solid, 431
solid, 431
semi-asphaltic, 55, 62, 72, 73, 409, 410, 778
settling of, 411
skimming of, 418
solid paraffins in, 410
solubility of, 54
steam-distillation of:
asphaltic oils, 412, 414, 415
non-asphaltic oils, 414
semi-asphaltic oils, 414
straight-running of, 414
topped, 418, 430
topping process of refining, 418
types of, 410
vacuum distillation of, 417, 418
varieties of, 409
weather-resistance of, 478
Petroleum asphalts, 409, 431
production of, 99, 103, 104
straight-run, 414
Petroleum coke, 416, 417
Petroleum distillates, 429
Petroleum fields, 410
Petroleum grease, 431
Petroleum jelly, 431
Petroleum naphtha, 429
solubility in, 964
Petroleum paraffin, 63, 416, 417, 431, 466
Petroleum pitch, 431, 525 (insert), 778
Petroleum residues, 414, 431
Petroleum resins, 466, 470
properties of, 470
recovery of, 470
solubility of, 470
uses of, 470
Petroleum solvents, 125, 516, 725
Petroleum spirits, 429
Petroleum tailings, 431
Petroleum tar, 525 (insert), 778
Petrologen, 277
Phase, 503
continuous, 503, 504
discrete, 503, 504
external, 503, 504
internal, 503, 504
Phenol-aldehyde resin, 487, 509, 542, 713, 721, 736, 749, 755
Phenolpech, 377
Phenols from:
asphalt, 210
cannel-coal tar, 336
coal tar, 346, 349, 366, 370 (insert)
peat tar, 308
Phenols from—*Continued:*
shale tar, 331
torbanite tar, 336
wood tar, 300
Philippines:
asphalt in, 153
ozokerite in, 134
Phonograph records, use of asphalt in, 557
Phosphoric acid, reactions of, 58, 79, 497, 509
Phosphorus, reactions of, 58, 80
Physical characteristics of bituminous substances, 780
Physical tests of:
bituminized fabrics, 1072
bituminous-solvent compositions, 1117
bituminous substances, 780
Phytocollite, 96
Phytosteryl, 1006
Pianzite, 96
Pigments, 497, 498, 499, 631, 724, 751, 1063, 1128
examination of, 1063, 1066
Pine oil, 296
Pine tar, 297
classification of, 57, 63
production of, 297
properties of, 298, 778
solubility of, 525 (insert)
word in:
Accadian, 2
Assyrian, 2
Greek, 2
Latin, 2
Sumerian, 2
Pine-tar pitch, 296, 297
classification of, 63
composition of, 300
distinguishing of, 298
production of, 299
properties of, 299, 778
solubility of, 525 (insert)
weather-resistance of, 300
Pinoline, 302, 303
Pintsch gas, 382
Pintsch-gas tar, 382, 384
Pipe-coatings, 104, 565
Pipe-dips, 491, 493, 535
Pipe-joints, 554
Pipes, 546, 557
asphalt, 713
Pipe-sealing compounds, 491, 493, 535, 535
examination of, 1017
Pipe-stills, 412, 417, 418
Pipe-wrappings, 538
Pissasphaltos, 2, 31, 34, 36
Pitch:
acid-sludge, 321, 323
amber-tar, 280
animal-oil-tar, 280

Pitch—*Continued:*
anthracene, 58, 376
anthracene-oil-tar, 281, 376
asphalt-tar, 281
bagasse-tar, 280
beechwood-tar, 300
beeswax-tar, 280
beet-residue-tar, 280
blast-furnace coal-tar, 371
bone-fat, 386, 396, 402, 405
bone-tar, 386, 407
brewers', 304
Burgundy, 304
candle, 386
carbol, 281
cheese, 189
Chemopeche, 58
cholesterol, 386, 399
classification of, 63
coal-tar, 336
coke-oven coal-tar, 371
cokey, 190
composition of, 54
cork-tar, 280
corn-oil, 386, 395
corn-oil-foots, 386, 395, 402, 405
cotton, 386, 395
cotton-oil, 386, 395
cotton-seed-foots, 386, 402
cotton-seed-oil, 386, 395
cotton-seed-oil-foots, 386, 395, 405
cotton-stearin, 386, 395
cresol, 58, 281
cumarone, 281
definition of, 58
distinguishing from asphalts, 456, 457
fat, 386
fatty-acid, 386
fermentation-residue, 280
Fettpech, 386
fluxing of, 479
fullers-grease, 386, 398
fusel-oil-tar, 281
garbage, 386, 397, 402, 405
gas-producer coal-tar, 371
gas-works coal-tar, 371
glance, 227, 236
glycerin, 408
gutta-percha, 58
hardwood-tar, 299
hydrogenation of, 58
immediate, 58
iron, 190
Karbolpech, 377
Kresolharz, 377
leather-tar, 280
lignin-tar, 280
lignite-tar, 310, 320
lignite-tar-sludge, 320, 321
linoleum-tar, 280

Pitch—*Continued:*
low-temperature coal-tar, 371
mineral, 44, 221
molasses-tar, 280
montan, 58, 137
montan-tar, 280
montan-wax, 137
naphthol, 58, 377, 486
naphthylamine, 281
oil-gas-tar, 378
origin of word, 1
oxygenated products from, 58
ozokerite, 58, 130
packing-house, 386, 402
palm-oil, 386, 402, 405
peat-tar, 305
petroleum, 431, 778
Phenolpech, 377
pine-tar, 296, 297
potato-residue-tar, 280
precipitation products, 58
presence of iron or copper in, 71
producer-gas coal-tar, 371
properties of, 54, 479, 778
rosin, 301
rubber, 58, 375, 400, 401
seaweed-tar, 280
seed-tar, 280
seek-oil, 398
sewage, 386, 397, 402, 405
shale-tar, 324
solubility of, 54, 525 (insert), 729
stearin, 386
stearin-wool, 386
straw-tar, 280
sulfite-cellulose-tar, 280
tall-oil, 280
tannin-tar, 280
tobacco-tar, 280
vegetable-oil-tar, 280
vinasse-tar, 281
Vosges, 304
water-gas-tar, 378
weather-resistance of, 479
wood, 297
wood-tar, 288, 299
wool, 386, 399
wool-fat, 386, 399
wool-grease, 386, 399, 402, 405
word in:
Accadian, 2
Arabic, 2
Assyrian, 2
Greek, 2
Hebrew, 2
Latin, 2
Sanskrit, 2
Sumerian, 2
Turkish, 2
wortzilite, see "wurtzilite asphalt"

Pitch-and-felt roofs, 368
Pitch bay, 367
Pitch coal, 143
Pitch coke, 369
  cellular, 370
Pitch cooler, 360, 367
Pitch fillers, 594, 603
Pitch grease, 346, 370
Pitch lake, 42, 146
Pitch oils, 346
Pitch resin, 369
Pitch still, 390
Pit tar, 289
Pittsburgh flux, 431, 452
Plaster-board, bituminous, 719
Plastic asphalt-cement, 744, 746
Plastic cements, 744
Plastic-cement, self-hardening, 746
Plastic compositions, 528, 744
Plasticity, definition of, 828
Plasticity tests, 828
  consistometer method, 840
    for bituminous-solvent compositions, 1118
  Moh's scale, 829
  needle penetrometer method, 830
Plasticizer, 752
Plastic range, 1154
Plastic-slate cement, 528
Plastic waterproofing, 704
Pliability of roofings, 1080, 1158
Pliability test, 1080
  mandrel method, 1080
  Reeve and Yeager method, 1080
Plumbing, early use of asphalt in, 24
Poise, definition of, 817
Poland, ozokerite in, 132
Polycyclic hydrocarbons, 990, 1011
Polymerization of bituminous substances, 72, 94, 455, 714, 1150
Pool, subterranean, 86
Porch roofs, 653
Porosity of felts and papers, 1107
Porosity test, 1107
Portugal, asphalt in, 212
Portuguese West Africa, albertite in, 270
Potato-residue tar, 280
Potato-residue-tar pitch, 280
Pot-head compound, 546
Pot still, 355
Powders, see "fillers"
Precipitation products, 58
Preformed joints, 551, 612
Preformed washers, 559
Premolded strips, 612
Prepared roofings, 641
Prepared-roofing shingles, 658
Prepared sheet-roofings, 44, 617
Preservatives:
  creosote, 47, 600
  wood, 47, 370 (insert), 600
Press for asphalt blocks, 596
Pressure-tars, 279, 424, 425, 427, 428
  asphaltic resins from, 471
  blown, 428, 434, 437, 438
  classification of, 63
  distillation of, 427, 437, 438
  fluxing of, 439, 479
  formation of, 435, 438
  free-carbon removal, 434
  mixtures with:
    coal tar, 439
    coal-tar pitch, 484
    coke-oven tar, 439, 484
    sludge asphalt, 465
  properties of, 435, 439, 778
  steam-distillation of, 427, 428, 437, 439
  used for producing:
    asphalt flux, 428
    blown asphalt, 428, 438
    emulsions, 438
    fuel oil, 428
    hydrogenation, 429
    residual asphalt, 428, 439
    residual oil, 433, 434, 439
    roofings, 438
  weather-resistance of, 479
  yields of, 437
Preventatives, dust, 575
Primary deposit, 87
Primer for:
  built-up roofing, 733
  flooring compositions, 742
  ship-enamels, 532
  waterproofing, 733
Priming coat:
  asphalt, 733
  creosote, 698, 733
Producer gas, 285, 313, 316, 348
Producer-gas coal-tar, 279, 285, 348
  classification of, 57, 63
  composition of, 348
  distillation of, 354
  production of, 279, 348
  properties of, 351, 778
  recovery of, 348
Producer-gas coal-tar pitch, 371
  classification of, 58, 63
  properties of, 371, 778
Producer-gas lignite-tar, 317
Producer-gas peat-tar, 307, 308
Production of:
  asphalt, 98
  asphalt brick-sidings, 109
  asphaltic pyrobitumens, 98, 102
  asphaltites, 98, 102, 103
  blast-furnace coal-tar and pitch, 107
  bone tar and pitch, 107

Production of—*Continued:*
coal tars, 106
coke-oven coal tar, 106
fatty-acid pitch, 107
floor-coverings, 111
gas-works coal tar, 106
gilsonite, 103
grahamite, 103
native asphalts, 98, 100, 102, 103
oil-gas tar, 106
ozokerite, 103
paving materials, 107
petroleum asphalts, 98, 99, 103, 104
roof-coatings and cements, 109, 110
roofings and shingles, 108, 109, 110
rosin pitch, 107
saturated felts, 109, 110
shale tar and pitch, 107
water-gas tar, 106
waterproofing fabrics, 109
wood tar, 106
wood-tar pitch, 107
wurtzilite, 103
Propane de-asphalting, 128
Propane refining, 126
Protective bodies, 66
Protective colloid, 503
Pug-mills, 506
Pumpherston retort, 325
Punishment, early use of asphalt in, 26
Purity of bituminous substances, 493
Putty, asphalt, 744
Pycnometer method for specific-gravity, 796
Pyridine, solubility in, 966
Pyrobitumen, 66
asphaltic, 262
associated minerals, 83
behavior on heating, 73
classification of, 52, 62
composition of, 54
definition of, 55
dehydration of, 114
effect of heat on, 72, 89
geology of, 82
mining of, 112
non-asphaltic, 65
occurrence of, 84
origin of, 54, 82
properties of, 54, 778
shipment of, 113
solubility of, 54
transportation of, 113
veins of, 84, 86
Pyrobituminous shales, 275
associated minerals, 277
characteristics of, 276
depolymerization of, 276
differentiation of, 275
dissolving in coal-tar pitch, 485
Pyrobituminous shales—*Continued:*
distillation of, 277
effect of heat on, 276, 279
fluxing of, 276
properties of, 268, 778
Pyrogenous, definition of, 52
Pyrogenous asphalts, 56, 63, 409, 478
classification of, 56
fluxing of, 478
properties of, 778
solubility of, 525 (insert)
weather-resistance of, 478
Pyrogenous distillates, 52, 63, 478, 525 (insert), 778
Pyrogenous residues, 52, 63, 478, 525 (insert), 778
Pyrogenous waxes, 409
classification of, 52, 56, 63
fluxing of, 478
properties of, 478, 778
solubility of, 525 (insert)
weather-resistance of, 478
Pyrolysis, 278
Pyropissite, 56, 128, 136, 312
Pyroretin, 96
Pyroxylin, see "cellulose-ester lacquers"

Qasat, 219
Quarrying, 112, 233
Quick-setting bituminous dispersions, 1132, 1138
Quinoline, 346

R. A. (refined asphalt), 570
Rag felt, 618
Ragusa asphalt, 209, 280, 593, 604, 609
distillation of, 209, 280
sawing into blocks, 211
use in mastic roofs, 211
use in pavements, 493, 604
weathering of, 211
Rail fillers, 615
Railroad ties, 557
Ready roofings, see "roofings"
Recycling stock, 425, 428
Red oil, 320, 321
Reeve and Yeager's breaking-point test, 886
Reeve and Yeager's pliability test, 1082
Refikite, 96
Refined asphalt, 570
Refined coal tar, 367, 370 (insert)
Refined paraffin wax, 431, 466
Refined scale wax, 466
Refined tar, 370 (insert)
Refining of:
asphalts, 114
bitumens, 114
bituminous substances, 114
bone grease, 396

Refining of—*Continued:*
bone tar, 408
coal tar, 44, 355, 365
corn oil, 395
cottonseed oil, 393
fatty acids, 387
garbage grease, 397
lignite tar, 318, 320, 321
native asphalts, 114
oil-gas tar, 383
ozokerite, 130
peat tar, 308
petroleum, 412
refuse greases, 395
sewage grease, 397
shale tar, 332
Trinidad asphalt, 187
vegetable oils, 393
water-gas tar, 383
wood tar, 292, 296
woolen-mill waste, 397
wool grease, 398
Reformed-gas tar, 383
Refractive index, 994
Repairing and patching pavements, 570 (insert)
Reservoirs, subterranean, 86
Residual asphalt, 418
acid value of, 457
asphaltic constituents in, 1009
California grades, 457
carelessly prepared, 454
chlorinated, 80, 458, 721, 752
classification of, 56, 63
coefficient of expansion of, 875
definition of, 431
distinguishing from:
asphaltites, 456
blown asphalts, 450, 456
native asphalts, 456, 457
pitches, 456, 457
fluxing of, 479
from asphaltic petroleums, 415, 453
from pressure-tars, 428, 429
from semi-asphaltic petroleums, 453, 455
grades of, 457
mixtures with coal tar, 485
nitrated, 486
properties of 452 (insert), 455, 458, 459, 479, 778
relation between softening-point and sp., gr., 449
removal of free-carbon from, 454
removal of undesirable constituents from, 454
separation from lubricating oils, 423
solubility of, 525 (insert)
straight-run, 414
sulfurized, 453
weather-resistance of, 458, 460, 479, 1162
Residual oil, 414, 418, 479, 1162
asphaltic constituents in, 1009
blown asphalts from, 433, 434
characteristics of, 432
classification of, 56, 63
coefficient of expansion of, 875
cut-back, 414, 433
definition of, 431
differentiating between, 432
fluxing of, 434, 478
from asphaltic petroleum, 432, 433, 436
from non-asphaltic petroleum, 414, 432, 433, 436, 478
from pressure-tars, 433, 434, 439, 478
from semi-asphaltic petroleum, 414, 432, 433, 436, 478
mixtures with gilsonite, 229
mixtures with grahamite, 250, 258, 1009
mixtures with pyrobituminous shales, 276
nickel in, 71
polymerization of, 72, 455
properties of, 432, 434, 435, 436, 478, 778
relation between softening-point and sp. gr., 449
saponification value of, 457
selective solvent process, 434
solubility of, 525 (insert)
straight-run, 414
voids in, 801
weather-resistance of, 478
Residual pitch, see "residual asphalt"
Residues, lignitic, 97
Residues, petroleum, see "petroleum residues"
Residues, pyrogenous, 52, 63, 478, 525 (insert)
Residuum oil, 334
Resilience, test for, 920
Resilometer, 912
Resin acids, 1001
Resinous bodies in coke-oven coal-tar, 346, 369
Resin, pitch, 369
shale, 334
Resins:
asphaltic, 470, 1008
coal-tar pitch, 369, 549
cumarone or paracumarone, 483, 487, 509, 530, 543, 721, 729, 749, 753
fossil, 721, 722, 723, 1130
petroleum, 466, 470
phenol-aldehyde, 487, 509, 542, 713, 721, 736, 749, 755
polyhydric-alcohol, 721
shale, 334
softening-point test for, 906
styrene, 722
synthetic, 721
urea, 732
vinyl, 722

Retinasphaltum, 96
Retinite, 96
Retort:
  beehive, 344
  Broxburn, 329
  coke-oven, 345
  construction of, 284
  continuously operating, 314, 324, 340
  gas-works, 340
  Henderson, 329
  horizontal, 340, 345
  inclined, 340, 341
  lignite, 314, 316
  peat, 307
  Pumpherston, 325
  Rolle, 314
  rosin, 302
  shale, 324
  vertical, 340, 344, 345
  wood, 290
Retort lignite, 312, 313, 314
Retort lignite-tar, 317
Retort peat-tar, 318
Revetment, bituminous, 616
Rhodesia, asphalt in, 226
Ring-and-ball softening-point test, 904
Rings, early use of asphalt for, 5
Road binders, 368, 431, 567, 570 (insert), 571
Road-mix pavements, 583
Road oil, 104, 431
  coefficient of expansion of, 875
Road tar, recovery of, 364
Roadway, compressed, first use in Paris, 48
Rochlederite, 96
Rock asphalt, 62, 70
  classification of, 56, 62
  comminution of, 121, 169, 499
  distillation of, 120
  word in:
    Arabic, 2
    Greek, 2
    Sanskrit, 2
    Sumerian, 2
    Turkish, 2
Rock-asphalt mastic, 45, 605
Rock-asphalt pavement, 590
Rock, bituminous, 56
Rocks, impregnated, 86
Rolle retort, 314
Roll-roofing, 646
  production of, 108, 109, 110
Roof-cements, production of, 109, 110
Roof-coatings, asphalt-fibrous, 109, 737
  production of, 109, 110
Roof copings, 555
Roofing or roofings, 617
  analysis of, 1072, 1090
  asphalt built-up, 686
  asphalt-fibrous coating for, 737
Roofing or roofings—*Continued:*
  asphalt-mastic, 609
  base-sheet for, 693
  bituminous compositions for, 491, 493
  blistering of, 1155
  boat-deck, 653
  built-up, 47, 642, 685, 686, 693
  cap-sheet for, 687, 693
  checking of, 1154
  color of, 1157
  composition of, 644, 648, 649
  concealed nailing of, 684
  cracking of, 1157
  decorative roll-roofings, 655
  diamond-print, 658
  discoloration of, 651
  discovery of, 44, 47
  durability of, 652, 1158
  dusting of, 1157
  fastening devices for, 682
  fillers for, 630
  fire-resistance of, 688
  first use in United States, 47
  freight-car, 620, 685
  granules for, 632, 644
  guarantees of, 652
  identification of, 652
  improving color of, 650
  lap-cement for, 683, 734
  laying of, 685
  manufacture of, 644, 654
  marginal edge of, 650
  mean effective life of, 1158
  metal cleats for, 683
  mildewing of, 640, 698
  multiple-layered, 653
  nails for, 683
  non-adhesive, 650
  locomotive, 653
  ornamental, 655
  packaging methods, 684
  paints for, 632, 653, 654, 734
  passenger-car, 653
  petroleum-tank, 655
  pliability of, 1080, 1158
  porch, 653
  prepared, 417, 641
  prevention of wrinkling, 651
  production of, 108, 109, 110
  railroad-car, 620, 653, 685
  recoating of, 652
  resistance to heat, 1086
  rub-test for, 1092
  sampling of, 773
  saturating of, 47, 634
  scalloped and serrated, 657
  single-layered, 641
  "square," 641
  standing-seams of, 684
  strength of, 1077, 1078, 1158

Roofing or roofings—*Continued:*
surfacings for, 632
tar, 44, 686
tensile-strength of, 1158
testing of, 1072
voids in, 801
weathering of, 652, 1153, 1167
weight of, 1075
Roofing asphalt, 104
Roofing cements, 693
Roofing cleats, 684
Roofing compounds, 491, 493
Roofing fabrics, 617
Roofing felt, 617, 642
Roofing flux, 431
Roofing granules, 632, 644
Roofing packages, 684
Roofing paints, 734
Roofing shingles, see "shingles"
Roofing "square," 641
Roofing waste, extraction of, 128, 554
use of, 553, 554, 613, 695, 745
Roof tiles, 554
Roofs, asphalt-mastic, 609
Roofs, tar-and-gravel, 47
Ropes, bituminized, 712
Rosin, 301, 302
destructive distillation of, 302, 303
detection of, 1016, 1130
dry distillation of, 301
electrical resistance of, 542, 543
extraction from wood, 297
fluxing of, 479
hardening of, 721
intermittent distillation of, 301, 302
properties of, 479
recovery of, 302
steam-distillation of, 301, 303
sulfurized, 304
vacuum distillation of, 302
weather-resistance of, 479
Rosin oil, 296, 302
blowing of, 297
blue, 303
brown, 303
green, 303
yellow, 302
Rosin pitch, 301
classification of, 58, 63
fluxing of, 479
production of, 107, 301
properties of, 303, 304, 479, 778
recovery of, 303
solubility of, 525 (insert)
sulfurized, 304
weather-resistance of, 304, 479
Rosin spirits, 296, 302, 303
Rubber blended with bituminous substances, 445, 453, 486, 487, 513, 541, 549, 613, 629, 722
Rubber latex, 487, 513, 613, 629, 694, 707, 712
Rubber oil, 483
Rubber pitch, 58, 375, 400, 401
Rubber substitutes, 482, 489, 491, 513, 541, 547, 1055
Rugs, asphalt-base, 695
Rumania:
asphalt in, 205
ozokerite in, 133
Russia:
asphalt in, 124, 151, 214
extraction with water, 124
gilsonite in, 236
ozokerite in, 133

Sakhalin, asphalt in, 152
Salvador, glance pitch in, 241
Sampling bituminous substances, 757
all-levels, 758
average, 758
blended substances, 762
borings, 760, 772
bottle-and-beaker, 763
bottom, 760
cements, 775
composite, 758
containers for, 757, 762
continuous, 760, 765
crude substances, 762
crushed fragments, 771
cylindrical tanks, 759
definitions of, 758
dipper, 760, 767
directions for, 760
drain, 760
emulsions, 775
fabrics, 773
grab, 760, 771
japans, 775
lacquers, 775
liquid substances, 769
lower, 758
markings of, 761
materials flowing by gravity, 767
materials pumped under pressure, 765
middle, 758
mixed, 760
paving materials, 773
pipe-lines, 765
place of manufacture, 763
point of delivery, 767
powdered materials, 771
precautions in, 760
purpose of, 758
refined substances, 762
semi-solid substances, 768
solid substances, 768, 771
storage tanks, 763
thief, 760, 769

Sampling bituminous substances — *Continued:*
upper, 758
varnishes, 775
volatile substances, 762
weighted bottle or can, 767
zone, 770
Sand-asphalt pavement, 590
Sand cores, 368
Santo Domingo, glance pitch in, 238
Saponifiable constituents, 1000
examination of, 1006
of fatty-acid pitches, 400, 402
separation of, 1004
Saponification value, 403, 451, 457, 1002
Sapropel, 91, 96, 306
Sapropelite, 306
Sapropelite coal, 306
Sarco, 446
Saturated burlap, 637
Saturated fabrics, 639, 641
Saturated felts, 634, 639, 642, 693
analysis of, 1072, 1088
production of, 109, 110
resistance to heat, 1085
tensile strength of, 1077
Saturated hydrocarbons, 65, 349
degree of mercuration, 1000
determination of, 995
diethyl sulfate test for, 999
dimethyl sulfate test for, 999
formolite reaction for, 999
solubility in sulfuric acid, 995
sulfonation residue, 995
Saturated woven fabrics, 639
Saturating capacity of felt and paper, 1110
Saturating compositions:
asphaltic, 626
coefficient of expansion of, 875
fire-resisting, 628
light-colored, 627, 640, 694, 706
tar, 626
Saturating composition for:
brake-linings, 713
clutch-facings, 713
conduits, 713
cords, 712
coverings for wires, 712
felt, 485, 634
flashings, 642
flooring, 693
insulating board, 718
insulating tape, 715
membrane waterproofing, 697
paper, 706, 707
plaster-board, 719
roofing felt, 368, 625, 642
roofings, 625, 634
ropes, 712
shingles, 625, 634
Saturating composition for—*Continued:*
stucco-base, 719
tacking-strips, 714
waterproofing fabrics, 642, 698
wood-blocks, 598
Saturating speed of felt and paper, 637, 1109
horizontal method of test, 1109
vertical method of test, 1109
Saturator:
asphalt, 636, 645
tar, 47, 635
Saxony, montan wax in, 136
Saybolt (Furol) viscosity test, 812, 845, 1135
Scale wax, 415, 416, 431, 466
crude, 415, 416
refined, 415, 416, 466
Scheereite, 46, 135
Schutte viscosity test, 823
Schwelkohle, 312
Scleretinite, 96
Scriptures, reference to asphalt in, 19
Sculpture, early use of asphalt in, 12, 13, 14
Seal-coat, 431, 578, 580, 583
Sealing-wax, 551
Seaweed tar, 280
Seaweed-tar pitch, 280
Secondary deposit of bitumen, 88
Seconds, gilsonite, 228, 229
Sedimentation, 122
Seed-hull tar, 280
Seed-husk-tar, 280
Seed tar, 280
Seed-tar pitch, 280
Seek oil, 398
Seek-oil pitch, 398
Seepages of asphalt, 84
Selects, gilsonite, 228, 229
Sélénitza asphalt, 29, 31, 33, 36, 151, 206
Selenium, reactions with, 80
Selenium oxychloride, solubility in, 524, 966
Self-hardening plastic-cement, 746
Semi-asphaltic petroleum, 55, 62, 72, 73, 409, 410, 778
classification of, 55
Semi-stable bituminous dispersions, 1132
Setting of bituminous substances, 1150
Settlement test for bituminous substances, 1135
Sewage pitch, 386, 397, 402, 405
Seyali, 2, 219
Seyssel asphalt, 43, 45, 46, 192, 604
production of, 194
Shale, pyrobituminous, 275
Shale oil, see "shale tar"
Shale paraffin, 63, 333, 334, 466
Shale resin, 334

Shales:
Albert, 331
albertite, 276
Arcadian, 331
asphalt-bearing, 275
asphaltic pyrobituminous, 275
bituminous coal, 276
cannel coal, 276
coal, 62, 94, 276, 277, 778
coorongitic, 331
dissolving in coal-tar pitch, 485
distillation of, 279, 324
Kimmeridge, 331
kukkersite, 331, 335
lignite and lignitic, 62, 94, 276, 277
Lothian, 331
mining of, 324
molybdenum in, 71
non-asphaltic pyrobituminous, 275
oil, 275
oil-bearing, 275
oil-forming, 275
Orepuki, 331
pyrobituminous, 275
Scottish, 324
torbanitic, 277
United States, 331
wurtzilite, 276
yields on distillation, 330, 331, 334
Shale tar, 279, 324
classification of, 57, 63
composition of, 331
cracking of, 429
dehydration of, 332
discovery of, 43
distillation of, 332
distinguishing of, 332
Henderson distillation process, 332
production of, 107, 279
properties of, 331, 778
recovery of, 329
refining of, 332
solubility of, 525 (insert)
yields on distillation, 333, 334
Shale-tar pitch, 324
classification of, 58, 63
discovery of, 43
production of, 107
properties of, 335
Shatter test for paving compositions, 1024
Shatter test for ship-enamels, 533
Sheathing papers, see "building papers"
Sheet-asphalt pavements, 570 (insert), 575, 589
earliest use of, 48, 49
Sheet-asphalt topping, 589
Sheet roofing, 617
Shim-strips, 714
Shingles, 658
analysis of, 1072
Shingles—*Continued:*
Dutch-lap, 663
fastening devices for, 682
fire-resistance of, 688
general features, 658
individual, 660
laminated, 660
laying of, 685, 688
molded, 554
multiple shingle-strips, 668
nails for, 683
packaging methods, 685
packing machine, 645
prepared-roofing, 658
production of, 108, 109, 110
reinforced, 660
resistance to heat, 1086
self-spacing, 662
specifications for, 659
strip, 668
surfacings for, 659
testing of, 1072
thatched, 668
triplex, 668
twin, 668
unit, 660
weight of, 1076
wooden, 660
Ship-enamel, 531
brittleness test, 533
covering power, 532
flow test, 533
primer for, 532
shatter test, 533
workability test, 533
Shipping methods, 113
Ships, early use for asphalt in, 40, 41, 42, 151, 191, 219
Shoe fillers, 553
Shoe soles, 45
Siberia:
ozokerite in, 134
Sicilian asphalt, 29, 33, 36, 209, 593
distillation of, 209
use in pavements, 593
Sicily, asphalt in, 29, 33, 36, 209
Sidewalk tiles, 598
Sidings, asphalt, 681
production of, 109
Sieburgite, 96
Sieving test, 1056, 1133
Sieves, 1057
Sintex pavement, 587
Sitosteryl, 1006
Skimming petroleum, 418
Slack wax, 415, 416, 467
Slaters' felt, 639
Slime, 19, 42
Slow-setting bituminous dispersions, 1132 1138

Sludge, acid, 460
Sludge asphalt, 58, 78, 79, 320, 333, 415, 460
asphaltic constituents in, 1009
blowing of, 440, 445, 464, 465
classification of, 56, 63
definition of, 431
distinguishing characteristics of, 462
fluxing of 464, 465, 479
from asphaltic petroleum, 415
from lubricating oils, 460, 461, 463
lead in, 71, 462
mixtures with:
blown asphalt, 465
gas-works coal-tar pitch, 464
pressure-tar, 465
properties of, 461, 462, 464, 479, 778
solubility of, 525 (insert)
sulfurized, 453
weather-resistance of, 465, 479
Soft asphalt, word in:
Greek, 2
Latin, 2
Softening, see "fluxing"
Softening-point, 895
compression method, 912
cube method, 908
for paraffin wax, 914
for petrolatum, 913
Kramer-Sarnow method, 898
lowering of, 482
of molded compositions, 1038
raising of, 80, 485
ring-and-ball method, 904
Soft flux, 606
Soft-wood distillation, 279, 295, 296
Sol, 502
lyophobe, 66
Soles of shoes, 45
Solidifying-point test, 889
relation to liquefying-point, 892
Solid paraffins, 381, 630
determination of, 992
in petroleum, 410
Solubility in:
acetone, 78, 300, 970, 971, 973
alcohol, 78, 300, 726
aniline, 966
benzol, 965
carbon disulfide, 951, 971, 973
carbon tetrachloride, 962
ethyl ether, 971, 973
petroleum naphtha, 964
pyridine, 966
selenium oxychloride, 524, 966
sulfuric acid, 995
sundry solvents, 970
Solubility tests, 525, 951
Solvent naphtha, 365, 370 (insert)
Solvent paints, 720
Solvents, 514, 725
aceto derivatives, 518
alcohols, 518
behavior of, 515
capacity of, 728
chemical, 517, 725
chlorine derivatives, 517
coal-tar, 45, 125, 365, 370 (insert), 514, 515, 522, 725
esters, 519
estimation in paints, 1123
ethers, 518
evaporation rates of, 726
examination of, 1128
flash-points of, 516, 948
for cellulose-esters, 751
function of, 514
ketones, 520
mixtures of, 126
nitrogen derivatives, 517
petroleum, 125, 516, 725
specific-gravity test for, 1127
sulfur derivatives, 125, 517
sundry, 125, 725
surface-tension of, 521
wood, 125, 516, 725
Sound-proofing felts, 714
South America:
albertite in, 269
asphalt in, 146, 182
glance pitch in, 241
grahamite in, 256
impsonite in, 272
Spain, asphalt in, 197, 213
Specifications for:
acid-proof enamel for concrete, 534
acid-resisting asphalt varnish, 749
acid-resisting paints, 738
aggregates for paving, 574, 576
asphalt-fibrous roof-coating, 737
asphalt for:
built-up roofs, 527
damp-proofing, 526
paving, 568, 570 (insert)
waterproofing, 526
asphalt mastic, 605, 609
asphalt priming-coat, 699, 734
asphalt-saturated felt, 642
asbestos, 642
rag, 642
asphalt varnish, 748
bituminous cements, 745
bituminous expansion-joints, 613
bituminous grout, 531
bituminous ship-enamel, 532
bituminous varnishes, 747
built-up roofing fabrics, 642
built-up roofs, 686
cap-sheet, 646

Specifications for—*Continued:*
coal-tar pitch for:
built-up roofs, 527
damp-proofing, 526
paving, 570 (insert)
waterproofing, 526
coal-tar saturated felt, 639, 642, 693
creosote preservatives for wood-blocks, 600, 601
creosote priming coat, 698, 733
expansion-joints, 613
flashings, 642
floor tiles, 555
granules for roofings and shingles, 644
insulating tape, 715, 716
lap-cement for roll-roofings, 683
nails for roofings and shingles, 683
paving aggregates, 574 (insert)
paving binders:
liquid, 568
semi-solid to solid, 570 (insert)
paving emulsions, 573
pipe-seal compound, 540
plastic-cement, 745
roofing:
adhesives, 629
coatings, 629
felt, 622
granules, 644
rags, 618
saturants, 627
roofings, 646, 688
shingles, 646
surfacings for built-up roofs, 686
tarred felt, 639, 642
waterproofing fabrics, 642
waterproofing oils for concrete, 562
wooden paving-blocks, 598
Specific-gravity, apparent, 789
Specific-gravity tests, 788
analytical-balance method, 799
apparent, 1070
bottle method, 795
bulk, 1068
hydrometer method, 793
of bituminized mineral aggregates, 1017
of bituminous-solvent compositions, 1117
of mineral aggregates, 1066
of solvents, 1127
pyknometer method, 796
Westphal balance method, 795
Specific-heat test, 870
Spermaceti, 543
Sphagnum, 94
Sponge asphalt, 565, 613, 1154
Spontaneous hardening of bituminous substances, 1150, 1154
Spreading-capacity test for solvent compositions, 1118
Springs of asphalt, 24, 26, 84
Sprungwax, 132
Stable bituminous dispersions, 1132
Stability test for bituminous dispersions, 1135
Stabilizing agents, 503, 511
Stair-treads, early use of asphalt for, 8
Stampfasphalt, 589
Stanolind, 446
Stanolite, 427
Statues, early use of asphalt in, 10, 11, 13, 16, 17
Steam distillation of:
coal, 348, 379
coal tar, 355, 359, 360
coal-tar pitch, 368
fatty acids, 388, 389, 390, 395, 396, 398
lignite tar, 319
montan wax, 137
ozokerite, 130
peat tar, 310
petroleum, 412, 415
pressure tar, 427, 428, 437, 438
rosin, 301, 303
shale, 324
shale tar, 332
soft wood, 295
wood tar, 293
wool grease, 398
Stearin pitch, 386, 525 (insert)
Stearin-wool pitch, 386
Stellarite, 268
Step-treads, 557
Still grease, 333
Stills, batch, 412
Stills for:
coal tar, 355
coal-tar pitch, 369
fatty acids, 389
lignite tar, 313, 314, 316
peat tar, 307
petroleum, 412, 417
rosin, 301
shale tar, 324
wood tar, 290, 293, 295
wurtzilite, 472
Stills:
Leinweber, 363
pipe, 417, 418
pot, 355
Sadenwasser, 363
tube, 353, 362, 412, 417
Still wax, 468
Stockholm tar, 297
Stone-backing, 733
Stone, bituminized, 546
Stone-coating test, 1137
Stone-filled sheet-asphalt pavements, 586, 589
Storage-battery boxes, 546
Storage of bituminous substances, 128, 354
Straight distillation, 414
Straight-run asphalt, 414

Straight-run coal-tar pitch, 367
Straight-run residual oil, 414
Straw tar, 280
Straw-tar pitch, 280
Streak, 785
Strength:
  bursting, 1078, 1104
  compressive, 1034
  flexural, 1036
  Mullen, 1078, 1104
  tearing, 1078
  tensile, 1033, 1077, 1104
Strength-factor, 705, 706
Strength of:
  burlap or duck, 1105
  felt, 1105
  molded compositions, 1033, 1034
  paper, 706, 1077, 1104
  roofing, 1077, 1078, 1158
  tape, 1086
  woven fabrics, 1105
String-felt, 624
Strips:
  premolded, 612
  shingle, 668
Strip-test for strength, 1106
Stucco-base, bituminous, 719
Stucco-base coating, 741
Stuffing grease, 399
Sub-floor-tar waterproofing, 531
Sub-microns, 804
Substitutes, rubber, 491, 513, 517
Sulfite-cellulose tar, 280
Sulfite-cellulose-tar pitch, 280
Sulfonation residue, 995
Sulfur, 68
  determination of, 956, 984
  reactions with bituminous substances, 79, 452, 486
Sulfur dichloride, reactions of, 79, 383, 453,549
Sulfur dioxide, reactions of, 77
Sulfuric acid:
  reactions of, 78, 401, 453, 460, 486
  solubility in, 995
Sulfuric acid and formaldehyde, reactions of, 79, 999
Sulfur trioxide, reactions of, 78
Sulfurized anthracene pitch, 376
Sulfurized asphalt, 58, 431, 452, 486
  chlorinated, 453
  definition of, 431
  discovery of, 452
  from sundry mixtures, 452
  properties of, 452
  vulcanizing agents proposed, 453
Sulfurized blown petroleum asphalt, 443
Sulfurized coal-tar pitch, 375, 527, 549
Sulfurized fatty-acid pitch, 401, 403, 721
Sulfurized lignite-tar pitch, 323
Sulfurized native asphalt, 452
Sulfurized oil-gas tar, 383
Sulfurized residual asphalt, 453
Sulfurized rosin, 304
Sulfurized rosin pitch, 304
Sulfurized sludge asphalt, 453
Sulfurized Trinidad asphalt, 453
Sulfurized water-gas tar, 381
Sulfurized wax tailings, 469
Sulfurized wurtzilite asphalt, 453
Sumerians, use of asphalt by, 5
Surface, aged indoors, 784
Surface-course of pavements, 578, 584, 588, 590
Surface mixtures for pavements, 570 (insert), 579, 589
Surface-tension, 857
  of asphalts, 484, 521
  of coal tars and pitches, 521
  of solvents, 521
  molecular, 859
  Nellensteyn's method, 857
Surfacings:
  bituminous, 578
  coarse, 634
  colored, 632, 633
  fine, 632
  granular, 632
  inorganic, 632
  of mineral matter, 632, 644
  of vegetable matter, 634
  organic, 634
  uncolored, 632
Susceptibility index, 845
Susceptibility, reducing, 486
Suspending agent, 503
Suspensions of bituminous substances, 503
Suspensions of coal-tar pitch, 367
Switzerland, asphalt in, 43, 195
Syria:
  asphalt in, 152, 216
  glance pitch in, 242

Tabbyite, 140
Tablets, early use of asphalt in, 14
Tackiness test for tape, 1087
Tacking-strips, 714
Tag flash-point test:
  closed method, 945
  open method, 948
Tall-oil tar, 280
Tall-oil pitch, 280
Tanning-residue tar, 280
Tannin-tar pitch, 280
Tape:
  adhesion test for, 1087
  electrical insulating, 714
  oven test for, 1087
  tackiness test for, 1087
  tensile-strength test of, 1086

Tar:
acid, 460, 463
amber, 280
animal-oil, 280
anthracene-oil, 280
Archangel, 297
asphalt, 280
bagasse, 280
beechwood, 299
beeswax, 280
beet-residue, 280
blast-furnace, 63, 279, 286, 347
blau-gas, 383, 384
bog-head-coal, 336
boiled, 292, 295
bone, 279, 386, 407
browncoal, see "lignite tar"
candle, 386
cannel-coal, 336
carbocoal, 350
carburetted-water-gas, 286, 378
classification of, 52, 63
coal, 336
coalite, 349, 350
coke-oven, 279, 344
coke-oven-lignite, 317
cold, 578
colloidal particles in, 971
composition of, 54
cork, 280
cracking of, 238, 239, 358, 376, 429
cracking-still, 435
definition of, 57
Delmonte, 350
distillation of, see "distillation"
effect of temperature on, 282
fermentation-residue, 280
first use on flat roofs, 44
fluorescence of, 781
fluxing of, 478
fuel-oil-gas, 383
fusel-oil, 280
gas-producer, 348, 351
gas-works, 279, 339
generator, 289, 318
goudron, 386
hardwood, 297
Kerzenteer, 386
leather, 280
lignin, 280
lignite, 279, 310, 321
linoleum, 280
low-temperature-coal, 348
low-temperature-lignite, 317
methods of production, 278
mineral, 59
molasses, 280
Mond, 350
montan-wax, 280
mound, 289

Tar—*Continued:*
oil-gas, 279, 286, 378, 382
oil-water-gas, 383, 384
origin of, 54
oven, 289
peat, 279, 305
petroleum, 525 (insert)
pine, 297
Pintsch-gas, 382, 384
pit, 289
potato-residue, 280
pressure, 279, 424, 425, 427, 428
producer-gas, 279, 285, 348
producer-gas-lignite, 317
producer-gas-peat, 308
production of, 280
properties of, 54, 778
raw materials used, 279
refined, 370 (insert)
reformed-gas, 383
retort, 289, 295
retort-lignite, 317
road, 364
seaweed, 280
seed-hull, 280
seed-husk, 280
shale, 279, 324
solubility of, 54, 525 (insert)
soluble, 293, 294
Stockholm, 297
straw, 280
sub-floor, 531
sulfite-cellulose, 280
surface-tension of, 971
tall-oil, 280
tanning-residue, 280
tobacco, 280
torbanite, 336
vegetable-oil, 280
vinasse, 280
water-gas, 279, 287, 378
weather-resistance of, 478
wood, 279, 288, 297
yields of, 281, 282
Tar-acids, 366, 991
contraction test for, 991
determination of, 991
liberation test for, 991
Tar-acid oil, 366
Tar-and-gravel roofs, 47
Tar bases, 346
Tar-concrete pavement, 570 (insert)
Tar extractor, 342
Tar fog, 284
Tar-macadam pavement, 46, 570 (insert)
Tar oil, 290
Tarpaulins, 46, 639, 641
Tarred-board, 45
Tarred-felt, 639, 642, 693
Tarred slaters' felt, 639

Tar roofs, 44, 47, 686
Tar saturants, 626
Tar saturator, 635
Tar still, 417
Tar tester, 819
Tasmanite, 270
Tataros asphalt, 124, 204
Tearing-strength test, 1078
Temperature scales, 1182
Tensile strength of:
  bituminized fabrics, 1077
  bituminous substances, 854
  felts, 1105
  increasing of, 487
  molded materials, 1033
  papers, 1104
  roofings, 1158
  tape, 1086
  woven fabrics, 1105
Tensometer, 854
Tents, bituminized, 639
Teredo-proof coverings, 620
Terminology, see "definition"
Tertiary bituminous mixtures, 489
Test, acid-resisting, 739, 1122
Testing of:
  bituminized fabrics, 1072
  bituminized mineral aggregates, 1044
  bituminous dispersions, 1132
  bituminous-solvent compositions, 1117
  bituminous substances, 776
Tests:
  chemical, 777, 973
  mechanical, 777, 809
  objects of, 776
  physical, 777, 780
  requirements of, 776, 777
  solubility, 777, 951
  thermal, 777, 863
Texaco, 446
Texas:
  asphalt in, 169
  grahamite in, 247
  ozokerite in, 135
Texas asphalt:
  blowing of, 170
  extraction with water, 123
  use in pavements, 169
Theories of petroleum origin:
  animal, 92
  inorganic, 89
  vegetable, 90
Thermal conductivity test, 863, 866
Thermal conductivity units, 865
Thermal expansion tests, 871
Thermal tests, 777, 863
Thermometer scales, 1182
Thickness factor, 705
Thickness test of:
  bituminized fabrics, 1077
Thickness test of—*Continued:*
  felt, 1103
  molded compositions, 1025
  paper, 1103
  roofing, 1077
  woven fabrics, 1103
Thixotropy, 1150
Threaded-felt, 624
Tiles:
  asphalt, 201, 598
  bituminated cork, 552
  floor, 201, 555, 598
  roof, 554
  sidewalk, 598
Tobacco tar, 280
Tobacco-tar pitch, 280
Topeka pavement, 587
Topped petroleum, 418, 430
Topping of petroleum, 418
Torbanite, 268, 336
Torbanite tar, 336
Torbanitic shales, 277
Total bitumen, see "solubility in carbon disulfide"
  erroneous use of expression, 55, 60
Transformer coils, 542, 546
Transportation methods, 113, 148, 185, 208, 354
Tree-surgery, use of asphalt in, 38, 513
Trinidad:
  asphalt in, 40, 67, 70, 81, 93, 182
  grahamite in, 256
Trinidad asphalt, 67, 70, 81, 93, 182
  coefficient of expansion of, 875
  colloidal nature of, 70, 93, 186
  composition of, 186
  dehydration of, 114, 187
  discovery of, 40
  first use for paving, 49
  mineral constituents of, 186
  mining methods, 112, 185
  mixtures with coal tar and coal-tar pitch, 484
  molecular weight of, 990
  nickel in, 71
  properties of, 188
  refining of, 187
  structure of, 990
  sulfurized, 452
Trinidad lake, 93, 182
Trinidad-lake asphalt, 182
Trinidad-land asphalt, 189
Trinkerite, 96
Tube heaters, 418
Tubes, bituminized, 546
Tube stills, 353, 362, 412, 417
Tunnelling, 112
Tunnels, early use of asphalt in, 21, 31
Turkestan:
  elaterite in, 263

Turkestan—*Continued:*
ozokerite in, 134
Turkey-in-Asia, asphalt in, 29, 220
Turpentine, 289, 295, 301
wood, 296
Turpentine substitute, 430
Twisting-point test, 918
Tyndall effect, 66
Tzacutli, 49

Ubbelohde liquefying-point test, 917
Uintaite, see "gilsonite"
Ultimate analysis, 68, 982
Ultimate working unit, 1060
Ultra-microns, 167, 803
Ultra-microscope, 66, 81
Ultra-microscopic examination of:
asphalts, 524, 802
tars, 524
Uncombined mineral matter, 70, 955
United States:
albertite in, 269
asphalt in, 139, 155
bituminous matter discovered in, 47
first pavement in, 48
gilsonite in, 230
glance pitch in, 240
grahamite in, 245
impsonite in, 271
ozokerite in, 134
production of asphalts in, 98
wurtzilite in, 264
Unsaponifiable constituents, 1001
examination of, 1006
separation of, 1005
Unsaturated hydrocarbons, 65, 337, 349
degree of mercuration of, 1000
determination of, 995
diethyl sulfate test for, 999
dimethyl sulfate test for, 999
formolite reaction of, 999
solubility in sulfuric acid, 995
sulfonation residue of, 995
Uranium in asphalts and asphaltites, 71, 240
Urteer, 349
Utah:
albertite in, 269
asphalt in, 140, 172
gilsonite in, 49, 230
glance pitch in, 240
ozokerite in, 134
wurtzilite in, 50, 264

Vacuum distillation of:
asphalt, 453
coal tar, 357
coal-tar pitch, 370
fatty acids, 389
lignite tar, 319
Vacuum distillation of—*Continued:*
montan wax, 137
petroleum, 417, 418
rosin, 301, 302
Vacuum flash-coils, 417
Vacuum impregnating compounds, see "saturating compositions"
Val de Travers asphalt, 43, 48, 195
use in pavements, 593, 604
Vanadium in asphalts and asphaltites, 16, 71, 240, 243, 259, 260, 273
Varnish kettle, 747
Varnishes:
acid-proof, 749
air-drying, 740
asphaltic, 747
baking, 755
bituminous, 746
electrical, 746, 753
flat or frosted, 747
long-oil, 748
manufacture of, 747
mat, 747
oxidizing of, 440
rubber surfacing, 749, 750
sampling of, 775
short-oil, 748
Vaseline, 431
Vaseline oil, 320
Vegetable fillers, 499, 613
Vegetable matter, surfacings of, 634
Vegetable oils, 479, 482 722, 723
Vegetable-oil tar, 280
Vegetable-oil-tar pitch, 280
Vegetable theories of petroleum origin, 90
Veins of bitumens, 86
Venezuela, asphalt in, 50, 146
Venezuela lake, 146
Ventura fluz, 431
Vermiculite, 555, 632, 718
Vertical retort, 340, 344, 345
Vinasse tar, 280
Vinasse-tar pitch, 280
Viscose, 542, 623, 650
Viscosity tests:
absolute, 817, 920
alternating-stress method, 825
Engler method, 811
equi-viscous temperature, 810
falling-ball method, 823
falling-coaxial-cylinder method, 826, 1150
float test, 820
for bituminous-solvent compositions, 1118
Hutchinson's method, 819
Saybolt (Furol) method, 812, 845, 1135
Schutte test, 823
Voids, determination of, 800, 1017, 1071
Volatile matter, 282

Volatility of bituminous substances, 477, 478
Volatility test, 920
Vosges pitch, 304
Vulcanization, 79
Vulcanized, see "sulfurized"

Walchowite, 96
Wall-board, bituminized, 717
Warfare, early use of asphalt in, 29, 30, 35, 38, 39, 40
Warrenite pavement, 586
Washers, preformed, 551
Water, determination in:
  bituminized fabrics, 1088
  bituminous dispersions, 1143
  bituminous substances, 973
  felt, 1103
Water, test for absorption of:
  bituminized fabrics, 1084
  bituminized mineral aggregates, 1018, 1029
  bituminous dispersions, 1139
  bituminous-solvent compositions, 1121
  bituminous substances, 785, 1150
  coal-tar pitch, 368, 698
  electrical insulating papers, 709
  molded compositions, 1029
  paving compositions, 1018
Water gas, 286
  carburetted, 286
Water-gas tar, 279, 287, 378
  carburetted, 286, 378
  classification of, 57, 63
  coefficient of expansion of, 875
  composition of, 381
  cracking of, 429
  dehydration of, 380
  emulsion of, 380
  production of, 106, 378
  properties of, 381, 778
  refined, 601
  refining of, 383
  sulfurized, 381
Water-gas-tar pitch, 378
  characteristics of, 385
  classification of, 58, 63
  coefficient of expansion of, 875
  distinguishing from:
    coal-tar pitch, 384
    oil-gas-tar pitch, 384
  fluxing of, 479
  properties of, 384, 385, 479, 778
  resistance to moisture, 385
  solubility of, 385
  weather-resistance of, 479
Water-japans, 755
Waterproofing:
  adhesive compounds for, 697
  asphalt for, 104, 526
Waterproofing—*Continued:*
  coal-tar pitch for, 368, 526
  decay-resistant, 698
  early use of asphalt for, 8, 10, 21, 23, 31, 35
  elastic method of, 696
  hydrostatic pressure in, 701
  integral, 560, 696, 704
  materials for, 697
  membrane, 639, 696, 700, 704
  membrane-and-plastic, 704
  plastic, 704
  primer for, 733
  protection of, 702
  rigid method of, 560, 696
  sub-floor-tar, 531
  surface coatings, 704
Waterproofing compositions, 491, 526, 560, 697
  for cement-mortar, 560
  for concrete, 534, 560
Waterproofing fabrics, 642, 697, 700, 703
  production of, 109
Waterproofing methods, 702, 704, 705
Waterproof membranes, 639
Wax:
  bayberry, 543
  bees', 543
  blister, 132
  blower, 132
  candelilla, 543
  carnauba, 543
  Chinese, 543
  crack, 132
  crude, 415, 416
  crude scale, 415, 416
  electrical resistance of, 542, 543
  fibrous, 132
  fluxing with bituminous substances, 484, 489
  fossil, 132
  hard, 132
  Japan, 543
  marble, 132
  mineral, see "mineral waxes"
  mixtures with:
    asphalts, 480
    gilsonite, 484
  montan, 132, 136
  paraffin, see "paraffin wax"
  pyrogenous, 409
  refined paraffin, 431, 466
  refined scale, 415, 416, 466
  scale, 415, 416, 431, 466
  sealing, 551
  slack, 415, 416, 467
  softening-point test for, 906, 914
  spermaceti, 543
  still, 468
  wool, 398

Wax distillate, 418, 424
Wax-stone, 130
Wax tailings, 416, 417, 466, 467
  classification of, 63
  coloring of, 469
  composition of, 468
  definition of, 431
  production of, 279, 467
  properties of, 468, 469, 478, 778
  sulfurized, 469
  uses as flux, 468, 478
  weather-resistance of, 478
Waxy-grease from coal-tar pitch, 370
Wearing-course of pavements, 581, 583
Weathering tests, 1147
  accelerated, 1161
  actual, 1153
  conductivity test for, 1169
  effect of fillers on, 1154
  extent of weathering, 1169
  modified accelerated, 1177
Weathering tests of:
  asphalt-filler mixtures, 1171
  bituminized fabrics, 1153, 1167
  bituminous-solvent compositions, 1158, 1167
  bituminous substances, 488, 1159, 1167
  roofings, 1153
Weatherproof insulated wire, 712
Weatherproof properties of bituminous substances:
  increasing of, 487
Weight of:
  bituminized fabrics, 1072
  desaturated fabrics, 1099
West Indies:
  glance pitch in, 237
  grahamite in, 245
Westphal-balance method of specific-gravity, 795
West Virginia, grahamite in, 48, 245
Wheelerite, 96
White spirits, 429
Wires, insulation for, 712
Wood:
  destructive distillation of, 288
  distillation of, 289, 290, 295
  extraction of rosin from, 297
  moisture content of, 288
  preservatives for, 47, 370 (insert), 600
  varieties of, 288
  volatile matter in, 282
Wood-block pavements, 47, 570 (insert), 598
Wood creosote, 290
Wood oils, 289, 292
Wood pitch, 297
Wood preservatives, 47, 370 (insert), 600
Wood tar, 279, 288, 297
  classification of, 63
Wood tar—*Continued:*
  composition of, 299
  cracking of, 429
  dehydration of, 293
  distillation of, 292, 293, 296
  distinguishing of, 298
  formaldehyde condensation product, 721
  hardening of, 301, 721
  production of, 43, 106
  properties of, 298, 778
  refining of, 292, 296
  solubility of, 298, 525 (insert)
  use by ancients, 29, 34, 43
  yields of, 289
Wood-tar pitch, 288, 299
  blown, 301
  chlorinated, 301
  classification of, 58, 63
  composition of, 300
  distinguishing of, 300
  fluxing, 301, 479
    with fatty-acid pitch, 301
  hardening of, 301
  production of, 106
  properties of, 299, 300, 479, 778
  solubility of, 300, 525 (insert)
  sulfurized, 301
  use by ancients, 34
  weather-resistance of, 300, 301, 479
Wood turpentine, 296
Wool dégras, 398
Wool-fat pitch, 386, 399
  blown, 440
Wool grease, 386, 398, 479, 1006
  distillation of, 398
  fluxing of, 479
  properties of, 479
  weather-resistance of, 479
Wool-grease pitch, 386, 399, 402, 405
  identification of, 406
  solubility of, 525 (insert)
Wool oil, 397, 398
Wool pitch, 386, 399
Wool wax, 398
Workability test for solvent compositions, 1118
Woven fabrics, 625, 698
  analysis of, 1072
  mildew prevention of, 640
  "number" of, 1103
  saturated, 639
  tensile-strength of, 1078, 1105
  thickness test for, 1103
  weight test for, 1103
Wrapping cloth, bituminized, 620
Wrapping papers, waterproof, 709
Wrinkle-finish japans, 755
Wurtzilite, 264
  characteristics of, 262
  classification of, 55, 56, 57, 62

Wurtzilite—*Continued:*
depolymerization of, 72, 472
discovery of, 50
metamorphosis of, 95, 262
properties of, 262, 778
production of, 102, 103
Wurtzilite in:
North America, 264
United States, 264
Utah, 264
Wurtzilite asphalt, 472
characteristics of, 472, 475
classification of, 56, 63
distinguishing from:
blown asphalts, 475
fatty-acid pitch, 475
fluxing of, 479
production of, 472
Wurtzilite asphalt—*Continued:*
properties of, 473, 474, 479, 778
sulfurized, 453
weather-resistance of, 475, 479
Wurtzilite pitch, 472
Wurtzilite shales, 276
Wyoming, asphalt in, 173

Xylene equivalent, test for, 783

Yellow grease, 396
Yellow rosin oil, 302
Yield-value, definition of, 828

Zephet, 2
Zietrisikite, 640
Zietriszit, 132
Zift, 2, 28
Zonolite, 555, 632

A Selection of Recent Van Nostrand Reference and Text Books on General Chemistry

Asphalts and Allied Substances. *By* HERBERT ABRAHAM, *President of The Ruberoid Co.; Asphalt Shingle and Roofing Industry.* Fourth Edition.

Catalysis—From the Standpoint of Chemical Kinetics. *By* GEORG-MARIA SCHWAB, *Professor Extraordinary of Chemistry of the University of Munich and* HUGH S. TAYLOR, *David B. Jones Professor of Chemistry, Princeton University and* R. SPENCE, *Lecturer in Physical Chemistry, Leeds University.*

Newer Methods of Volumetric Chemical Analysis. *By* PROFESSOR RALPH E. OESPER, *Department of Chemistry, University of Cincinnati.* Translated from the second revised and extended German Edition.

Reagent Chemicals and Standards. *By* JOSEPH ROSIN, *Chief Chemist and Chemical Director of Merck & Co.* $6.00

Colorimetric Method of Analysis. A general treatment of the entire subject. *By* FOSTER D. AND CORNELIA T. SNELL, *Consulting Chemists and Chemical Engineers.*
Volume I. $9.50
Volume II.

Colloid Chemistry. Principles and Application. *By* JEROME ALEXANDER, *Consulting Chemist and Chemical Engineer.* Fourth Edition. $4.50

A Manual of Industrial Chemistry. *By* ALLEN ROGERS, *Dept. of Chemistry, Pratt Institute, Lecturer on Ind. Chem., Columbia University.* (In collaboration with eminent specialists.) Two Vols. (Not sold separately.) $13.00

Van Nostrand's Chemical Annual. Rewritten Seventh Issue, 1934. *Edited by* JOHN C. OLSEN, *Professor of Chemical Engineering, Polytechnic Institute, Brooklyn, N. Y., Formerly, Sec. and Pres., Amer. Inst. Chem. Engrs.* Flexible Fabrikoid. $5.00

Prescott and Johnson's Qualitative Chemical Analysis. *By* ROY K. MC ALPINE AND B. K. SOULE, *University of Michigan.* $4.50

Elementary Quantitative Analysis: Theory and Practice. *By* HOBART H. WILLARD, *Professor of Anal. Chemistry, Univ. of Mich. and* N. HOWELL FURMAN, *Professor of Chemistry, Princeton University.* Second Edition. $3.25

Organic Chemistry. *By* FRANK C. WHITMORE. *Dean, School of Chemistry and Physics, and Research Professor in Organic Chemistry, Pennsylvania State College.*

General Chemistry. *By* THOMAS P. MC CUTCHEON, *Professor of Inorganic Chemistry, University of Pennsylvania,* HARRY SELTZ, *Assistant Professor Physical Chemistry. Carnegie Institute of Technology, and* J. C. WARNER. *Associate Professor of Electro-Chemistry, Carnegie Institute of Technology.* Second Edition. $3.50

Inorganic Chemistry for Colleges. *By* WILLIAM FOSTER, *Russell Wellmann Moore Professor of Chemistry, Princeton University.* Second Edition. $3.90

Standard Methods of Chemical Analysis. *Edited by* WILFRED W. SCOTT. (IN COLLABORATION WITH RENOWNED SPECIALISTS.) Fourth Edition. Two Vols. (not sold separately). $12.00

Modern Physics. A Second Course in College Physics. *By* G. E. M. JAUNCEY, *Professor of Physics, Washington Univ., St. Louis.* Second Edition. $4.00

A Treatise on Physical Chemistry. *Edited by* HUGH S. TAYLOR, *David B. Jones Professor of Physical Chemistry, Princeton University.* A cooperative Effort by a Group of Physical Chemists. Second Edition. Two Volumes (not sold separately). $15.00

Elementary Physical Chemistry. *By* HUGH S. TAYLOR AND H. AUSTIN TAYLOR, *Assistant Professor of Physical Chemistry, New York University.* Second Edition. $3.75

An Introductory Course in Physical Chemistry. *By* WORTH H. RODEBUSH, *Professor of Physical Chemistry, Univ. of Illinois; and* ESTHER K. RODEBUSH. $3.75

Van Nostrand books sent on approval to residents of the United States and Canada. General Catalog sent free on request.